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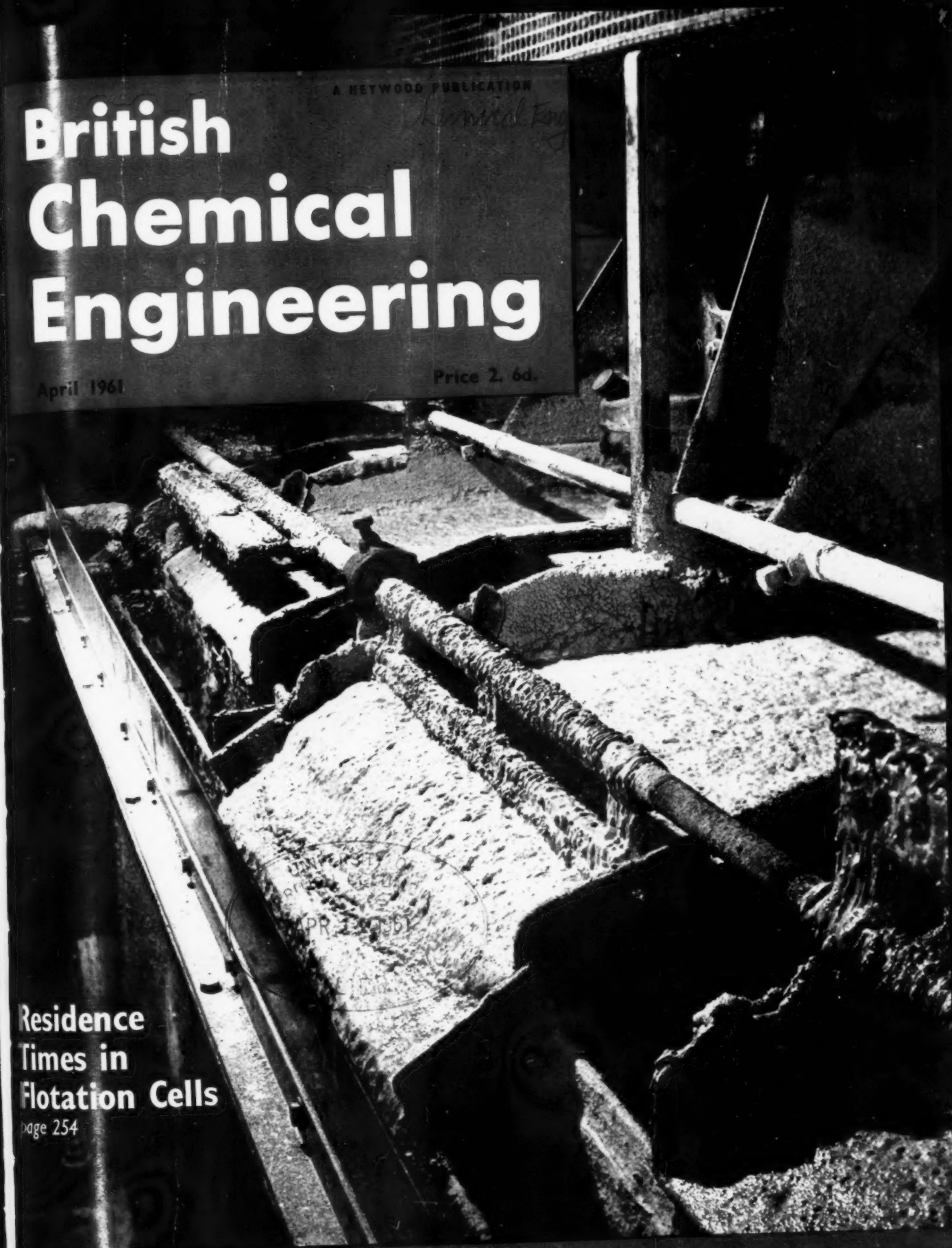

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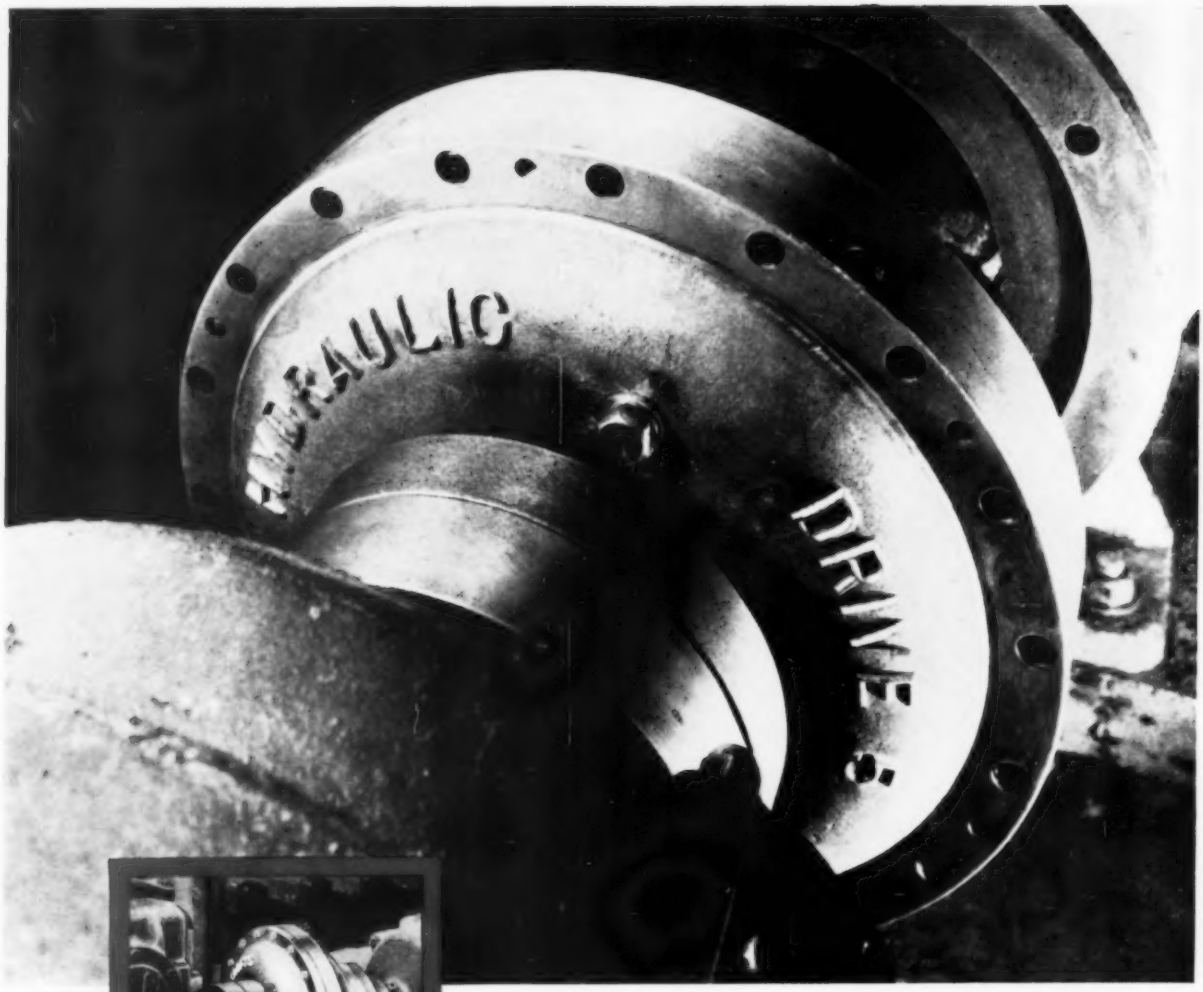

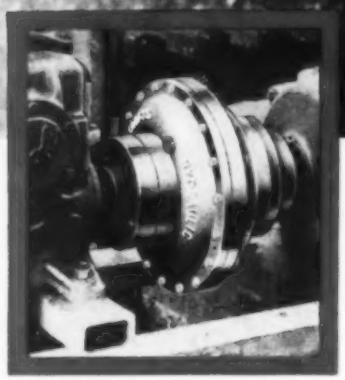

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Corrosion Snags in Gas Purification

CORROSION is one of the biggest problems in plants which use aqueous diethanolamine for removing H_2S from refinery gases. One of the components of plants using this process which suffers seriously is the reboiler of the regenerating section of the plant, the attack often being concentrated, in the case of kettle type reboilers, on the steel tubes as a result of hot spots at the baffles.

How to improve the resistance of a plant to this kind of corrosion has been outlined recently by K. L. MOORE, a corrosion engineer of the Tidewater Oil Company, Delaware, U.S.A. He found that he could virtually eliminate localised attack on the reboiler tubes by de-superheating the steam, operating at as low a steam pressure as possible, draining the tubes free of condensate and increasing the liquor height above the tubes. By these measures he was able to reduce the attack to a general thinning of the tubes which he found to be a function of the solution contamination resulting from decomposition of the diethanolamine and from substances extracted from the refinery stream.

Not the least interesting feature of MOORE's approach is his readiness to look for process modifications to reduce corrosion. He found, for example, that equipment was liable to suffer attack if the diethanolamine was richly loaded and gives a figure of 0.34 mols of acid gas per mol of "free" diethanolamine which should be observed if corrosion is to be avoided. If this loading is to be exceeded then one should use a higher concentration of diethanolamine, a higher circulation rate or one should revert to monoethanolamine.

Not all the corrosion problems referred to by MOORE were tackled in this way. Some required simply the use of other materials of construction. This applied to steel yokes (securing the bubble caps to the regenerator trays) which suffered from stress corrosion cracking. Replacement by stainless steel (Cr 17·5-20, Ni 8-11, Mn 2 max) and similar measures were taken with the main circulation pumps which had cast iron impellers and which failed from corrosion erosion in ten months. The use of the same alloy for these impellers provided a good solution.

"*Corrosion*" October 1960, 16, 12, 111.

Reaction Engineering Proceedings

TO gain an insight into the special character of chemical reaction engineering one could not do better than to refer to the complete proceedings of a conference reported in this Journal many months ago. This branch of chemical engineering is concerned in particular with the interaction of physical and chemical factors in chemical reactors and can be looked upon as a junction of the unit operation and unit process concepts.

In the proceedings of the event to which we refer, the Chemical Reaction Engineering Conference held in Amsterdam last Spring, many stimulating ideas are to be found among the many aspects of the field embraced by the event. And some timely advice was given about the risk of overestimating the usefulness of mathematical techniques for situations where the basic data is scanty.

This should not give the impression, however, that the event was academic in character; on the contrary, a high proportion of papers were devoted to design and scaling-up. While their mention is in no wise a reflection upon the others, two papers which seem especially interesting were devoted to the catalytic conversion of SO_2 and the design of ion exchange equipment. In the first, SCHWALB and SCHYTIL put forward the view that film and pore diffusion of oxygen affect the reaction rates even at high conversions, a proposition with which, judging from the discussion, other experts in the field do not agree. For the design of ion exchange equipment BECKER-BOOST describes a procedure which requires the experimental determination of the relations between the loading on an exchanger and the residence time of the liquid and its concentration as preliminary stages, in the dimensioning of a commercial unit.

If criticism is to be levelled it should surely be at certain features of the volume containing the proceedings.* Many of the translated English summaries could have been better edited and, in view of the translation resources possessed by the publishing company, there seems little justification for not having separate English translations of the papers given in other languages. In view of the expense of this volume (£8) such shortcomings, however trivial, are unexpected.

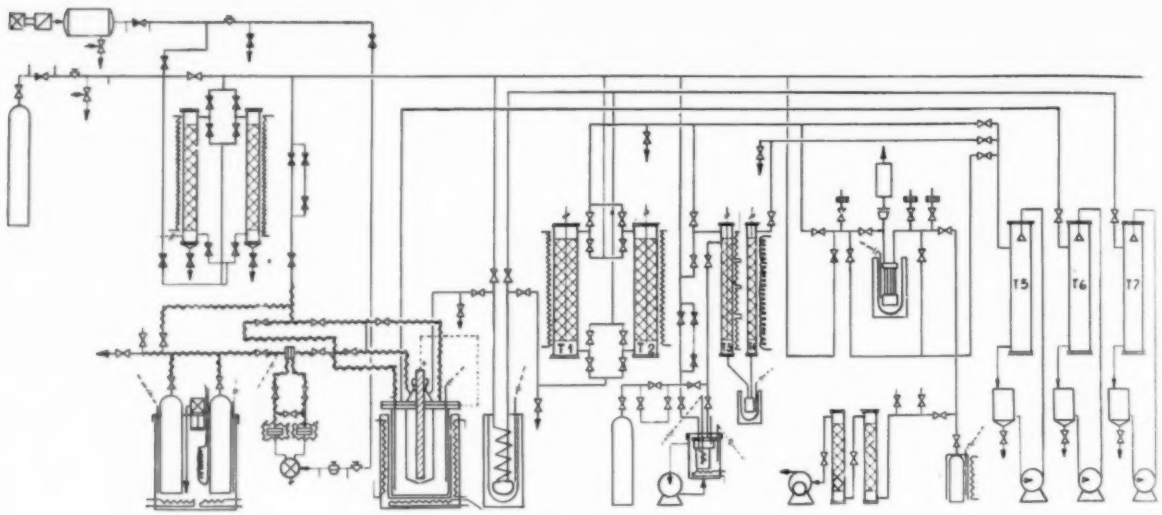
*Chemical Engineering Science, 1961. Vol. XIV

Silicon Carbide in the Ascendant

YET another unusual material with exceptional properties has appeared upon the scene to aid the chemical engineer in his search for materials capable of withstanding highly aggressive environments where conditions of corrosion, erosion and high temperature occur simultaneously.

This new material, developed by the Carborundum Company, is a dense, impermeable, self-bonding silicon carbide. It is capable of being formed into articles of both simple and complex shape such as tubing, grids, and to precise dimensions. Exceptional properties are hardness which is some 50 per cent greater than that of tungsten carbide, high thermal conductivity 600 btu/hr. ft^2 °F in. at 800°F, and a maximum oxidising temperature of 3000°F, the latter representing a considerable improvement on impervious graphite.

Applications illustrative of the exceptional resistance of this ceramic include its use as the lining material of a rotary kiln for processing uranium ores at 700°C in an atmosphere of ammonia, for the tubes of heat exchangers handling abrasive slurries containing 50 per cent nitric acid, for pipelines carrying chlorine at temperatures in the range 400-600°C; another interesting application is for orifice plates metering 95 per cent sulphuric acid at 200°C, with a complete absence of either corrosion or erosion. A number of other successful uses have been reported, such as for spray nozzles in spray driers handling abrasive slurries, valve components, thermocouple sheaths and rotating mechanical seals.



Laboratory Plant for Fluorine and Fluorine Halides

FLUORINE and its compounds are of great interest to the nuclear chemist, in particular uranium hexafluoride, which has applications in isotopic gaseous diffusion processes and in re-processing of fuels, dry decontamination, and uranium extraction from rich ores. To produce uranium hexafluoride, a source of elemental fluorine, or fluorine chloride or bromide, is required. An experimental laboratory plant for the production of fluorine and fluorine halides by electrolysis of melted potassium hydrogen fluoride has recently been put through preliminary tests in Italy, at the Centro di Studi Nucleari, Ispra.*

In this apparatus—which has a capacity of about 700 grams per day of fluorine—developed by G. CAMOZZO and S. PIZZINI, fluorine is produced in an electrolytic cell containing melted KHF_2 and maintained at a temperature of 250°C. The anode consists of a 300 mm high carbon bar and the body of the cell comprises the cathode. The bath concentration is kept constant by introducing, as required, a mixture of hydrofluoric acid gas and nitrogen. The gaseous HF, which flows through pipelines maintained at 80°C, is obtained for storage vessels maintained at a slightly raised temperature.

Hydrogen generated in the cell is passed direct to an alkaline scrubbing tower T_6. The fluorine, which contains some HF as an impurity, passes to a purification system comprising a condenser and heated absorption towers T_1 or T_2, filled with sodium fluoride pellets. The condensing stage, at minus 80°C, does not completely eliminate the HF. In the absorption stage, the reaction $NaF + HF = Na\,HF_2$ occurs, and at 100°C the HF is practically eliminated. The fluorine can either be utilised—compressed or reacted with bromine or chlorine— or it can be passed to a spray tower T_5 where it is absorbed by alkaline solutions. The hydrofluoric and nitrogen mixture which comes either from the condenser, or from the absorption towers T_1 and T_2 during regeneration of the sodium fluoride (by heating to 275°C), is fed to another absorption column T_7.

For compression of the fluorine, liquefaction followed by successive evaporation in cylinders has been adopted as the simplest and cheapest method. Cylinders and fluorine condenser are in Monel metal, but to keep costs down pipes and valves are in steel.

Chlorine or bromine trifluoride can be directly synthesised. A flow of nitrogen previously saturated with bromine, or a flow of chlorine, is introduced into tower T_3 where it reacts with the fluorine. The compounds are collected in a vessel immersed in a Dewar flask. While tower T_3 is in operation, tower T_4 acts as a reflux condenser and the excess of fluorine is discharged to the spray tower T_5. Tower T_4 can also be used for a preliminary distillation of the raw product.

* *Energia Nucleare*, 1960, 7, 12, 849.

Heat Transfer to Superheated Steam in Round and Rectangular Channels

PROPOSALS for superheating nuclear reactors have highlighted the need for more specific insight into the mechanism of heat transfer to flowing superheated steam. In practice, it is desirable to arrange flat-plate fuel elements to form thin rectangular channels, and this necessitates consideration of the influence of flow channels with high aspect ratios. With these objectives in view, an investigation has been carried out by J. HEINEMAN at the Argonne National Laboratory* on the convection heat transfer to superheated steam at intermediate pressures over a wide range of Reynolds number on both circular and rectangular tubes.

The round tube was 0.333 in. I.D. by 12 in.; the thin rectangular channel, 0.047 in. wide, aspect ratio, 26.6:1, and 12 in. long. The experiments encompassed a Reynolds number range from 20,000 to 370,000, pressure range from 300 to 1500 psia, inlet superheat from 5 to 160°F, and film temperature differences from 30 to 550°F.

From the round channel experiments, it was found that convection coefficients could be predicted within ± 10 per cent, using the following equations and values of thermal conductivity and viscosity presented by HEINEMAN in tabular form:—

$$\frac{hD_e}{k_f} = 0.0157 \left(\frac{GD_e}{\mu_f}\right)^{0.84} \left(\frac{c_p\mu}{k}\right)_f^{1/3} \left(\frac{L}{D_e}\right)^{-0.04}$$

for $\left(6 < \frac{L}{D_e} < 60\right)$

and

$$\frac{hD_e}{k_f} = 0.0133 \left(\frac{GD_e}{\mu_f}\right)^{0.84} \left(\frac{c_p\mu}{k}\right)_f^{1/3}$$

for $(L/D_e > 60)$

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Where

D_e = equivalent diameter = 4 × flow area/inside perimeter, in.; the remaining symbols have the meaning usually assigned to them in heat transfer literature with L, the length of channel measured in metres.

The type of unheated entrance section preceding the heated length of channel influences the heat transfer results markedly. The first of the above equations is applicable when this precedent section is 16 diameters in length. It may be safely used (with respect to limiting surface temperatures) for shorter unheated sections and should closely apply for a more fully developed velocity profile (unheated $L/D_e > 16$), because of the relatively weak influence of L/D_e.

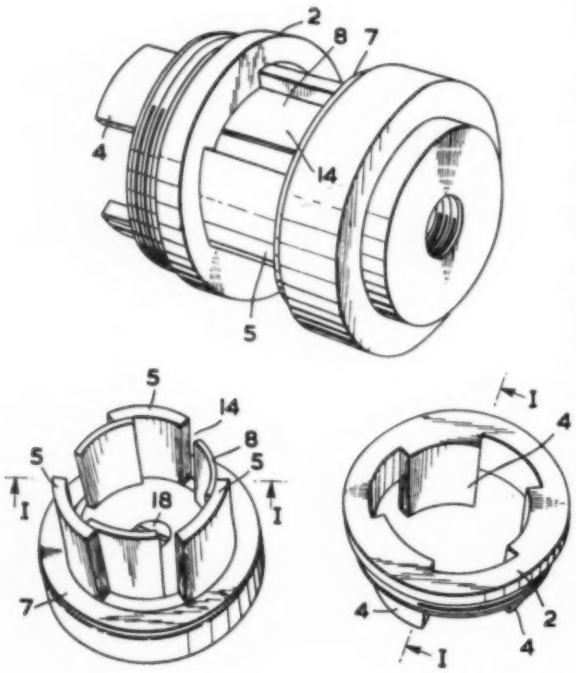
In the thin rectangular channel experiments, the vena contracta caused by the sharp-edged entrance condition influenced the heat transfer coefficient to a length of approximately 75 equivalent diameters, including the unheated entrance length of 13 diameters. There was no observable difference between the rectangular data and the round channel correlation in the L/D_e range from 60 to 121.

* ANL—6213 Reactors General. AEC Research and Engineering Report, Argonne National Laboratory Illinois, 1960.

A Novel Control Valve

THE control of fluid flow is ever a subject for the inventors' ingenuity, a remark borne out by the recent device emanating from the U.K.A.E.A. This is a valve designed by two engineers of the authority's Capenhurst works, which provides a good range of flow characteristics; the heart of the device is its specially-shaped seating and closure sections which can readily be used with some existing commercial valve bodies. Both of these carry interleaving tongues (4 and 5 in diagrams), but can slide between each other in such a way that the liquid is made to pass through ports defined by them.

Adjustment of flow characteristics is provided by a movable shutter over the spaces between the tongues on the closure section. This shutter normally rotates but can also be made to slide axially. In this case one can keep the ports closed during the earlier part of the travel of the closure section, or have a constant flow rate during the latter part of the travel.



A notable feature is that either type of shutter can be profiled to give a wide variation in flow rate characteristics. For example, a rectangular profile offers substantially linear characteristics, while a logarithmic profile can provide a logarithmic pattern.

Sealed closure is obtained by means of a sealing ring 7 mounted on the closure section which co-operates with the seating section 2; and the valve is made leak-tight through the use of a flexible metallic bellows sealed at one end to the valve body and at the other to a disc integral with the valve spindle. Referring to the diagram, ring 2 screws into the exit port of the valve and flow is through spaces 14 and out through the port into which 2 is screwed.

The apertures 14, determining the flow for any particular position of the valve during operation, are obtained from adjustment of the position of part 8 relative to part 5.

The fact that this valve can readily be adapted for automatic control and can be adjusted by conventional electrical or fluid operation without interfering with the automatic system controls, is a further feature in its favour.

Fusion Pots on the Way Out?

A GREAT deal of caustic soda is sold as concentrated solution containing 50 per cent or 70-74 per cent hydroxide and rather less is supplied as anhydrous solid. Apart from the convenience of transportation in liquid form production of the solid base is not encouraged by the difficulties of dehydration. The final stage of this part of the caustic soda process is carried out in cast iron fusion pots which have to be heated at something like 550°C before the final dehydration is achieved. This part of the process is batchwise—a batch usually being 12-18 tons,—and it is not surprising that the life of the pots is comparatively short; a life in excess of 200 days appears to be exceptional.

Among the other disadvantages of the fusion process are the contamination of the hydroxide by iron necessitating treatment with sulphur, and the fact that about 5 per cent of the output has to be either discarded or reprocessed.

To overcome these shortcomings a number of proposals have been made in the past such as the spray evaporation, filmwise evaporation using Dowtherm as the heating medium, and the azeotropic distillation of the strong liquor. Treatment of the liquor with anhydrous liquid ammonia has also been proposed. A partial pressure process was devised many years ago by OTHMER, JOSEPH and JACOBS and a recent paper by I. PETEU* seems to indicate a revival of the process.

PETEU confirms the suitability of petroleum fractions for the azeotropic agents, a useful property being the range of vapour pressures which covers the range of boiling points of 50 per cent solution down to very nearly anhydrous caustic. According to him this process requires a third the amount of fuel required by the fusion pot method and any concentration of caustic solution can be worked up to give 98 to 98.5 per cent NaOH for the azeotropic distillation. It is generally reckoned that Dowtherm is superior to steam as a heating medium and the installation of the Dowtherm vapouriser does not detract from the economics of the process; utilities costs, in fact, are approximately half those of the fusion pot process.

There seems to be a distinct prospect of a production unit operating this process being installed in Rumania. If so it will be interesting to see whether the pattern of caustic soda supply will be in anyway modified by its adoption.

* ING. I. PETEU, *Revista de Chimie*, vol. II, No. 12. December 1960, 694-697.

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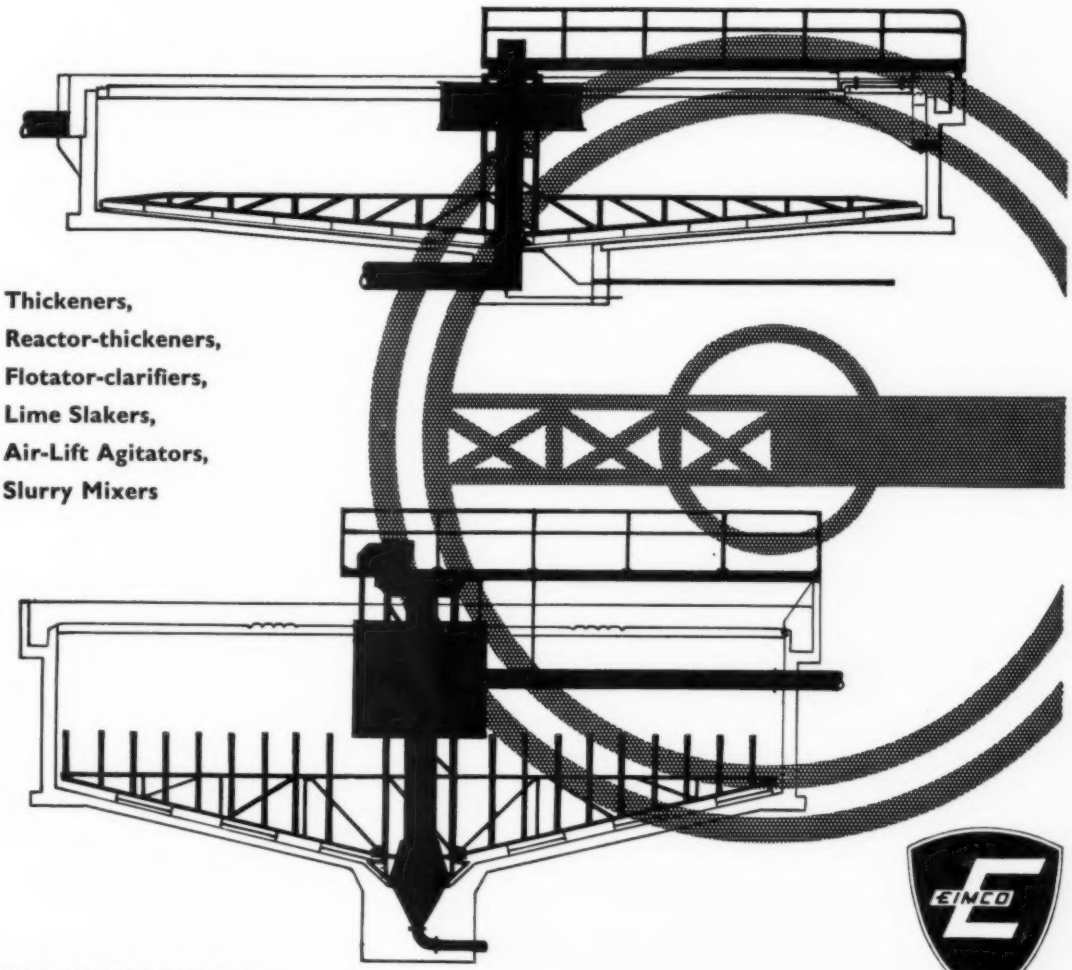

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P.2699

Materials of Construction Symposium

A ONE day symposium is to be held in the Chemical Engineering Department of the University, Edgbaston, Birmingham, 15, on Wednesday, April 12, 1961.

Two sessions, one for the morning the other for the afternoon have been arranged. During the former session which is to commence at 9.30 a.m. under the chairmanship of PROFESSOR F. H. GARNER, O.B.E., the following papers will be presented:

"Fabrication and Properties of Some of the Newer Constructional Metals," by DR. N. P. INGLIS (I.C.I. Metals Division).

"Recent Developments in Wrought Corrosion Resistant Nickel-Base Alloys," by MR. E. WARDE, Esq. (Henry Wiggin Co. Ltd.).

For the afternoon session, which commences at 2 p.m. MR. K. M. CURWEN will take the chair and the following papers will be read:

"Plastics in Chemical Engineering and Building," by S. SLANEY, Esq. (Shell Chemical Co. Ltd.).

"Glass Fibre Wound Laminates," by DR. F. F. JARAY (Consultant).

"Recent High Temperature Ceramics," by DR. A. T. GREEN (Consultant).

Further details and applications for Registration forms should be made to: T. R. BOTT, Esq., Hon. Secretary, Midlands Branch, The Institution of Chemical Engineers Chemical Engineering Department, The University, Edgbaston, Birmingham, 15.

Supercritical Grinding

ONE of the first fruits of the decision to make D.S.I.R.'s new Warren Spring Laboratory at Stevenage a centre of information and research in the field of mineral processing is the recent issue of two notes dealing with some important aspects of mineral processing.

The first deals with a subject which received attention a few years ago in this Journal, supercritical grinding. The possibilities of operating ball or rod mills at supercritical speeds is currently arousing much interest, and research is currently in progress in the United Kingdom, Australia, South Africa, the United States and a number of European countries. In the note this work is briefly summarised, and areas of investigation as yet uncovered include (1) the relationship between mill capacity and mill speed, (2) the relationship between power needed and mill speed, (3) the optimum amount of grinding media at different speeds, (4) the rates of wear of mill liners and grinding media, (5) the effects of different types of mill liners on mill operation and capacity, (6) the effects of the characteristics of the grinding media, (7) the effects of the type of mill e.g. high or low discharge, (8) the most suitable types of mill feeders for use at high speeds, (9) the extent to which loss of mill capacity in autogeneous grinding can be offset by operating at high speeds, (10) the effect of high mill speeds on the size distribution of the product, (11) the mechanism of the grinding process at high speeds.

Cyclones in Mineral Processing

THE second note reviews current information on the theoretical and practical aspects of the design and performance of cyclones with the main emphasis on hydraulic cyclones. Summarising the findings on liquid/solid separations, it is concluded that, while a cyclone must be designed to meet a particular application rather than the conditions adjusted to match a standard cyclone, there are nevertheless advantages to be gained by accepting a standard geometry. It provides a framework from which a specific design may be derived by a limited amount of test work. Three variables which do not appear to be adequately investigated are (1) feed section geometry (2) cone angle and (3) viscosity and pulp density effects.

As regards gas and liquid separations, gas/solid separations are well understood. Gas/liquid separations are not yet of much importance in the mineral industry, whereas liquid/liquid separations are of increasing interest for extraction techniques in hydrometallurgy and with the possibility of heavy liquid recovery units in ore treatment plants. The work carried out on liquid/liquid systems has defined the effects of process variables qualitatively; they should now be placed on a quantitative basis.

D.S.I.R. Information Note 1. "Supercritical Grinding."
D.S.I.R. Information Note 2. "Cyclones in Mineral Processing."

Vibration for Improved Mass Transfer

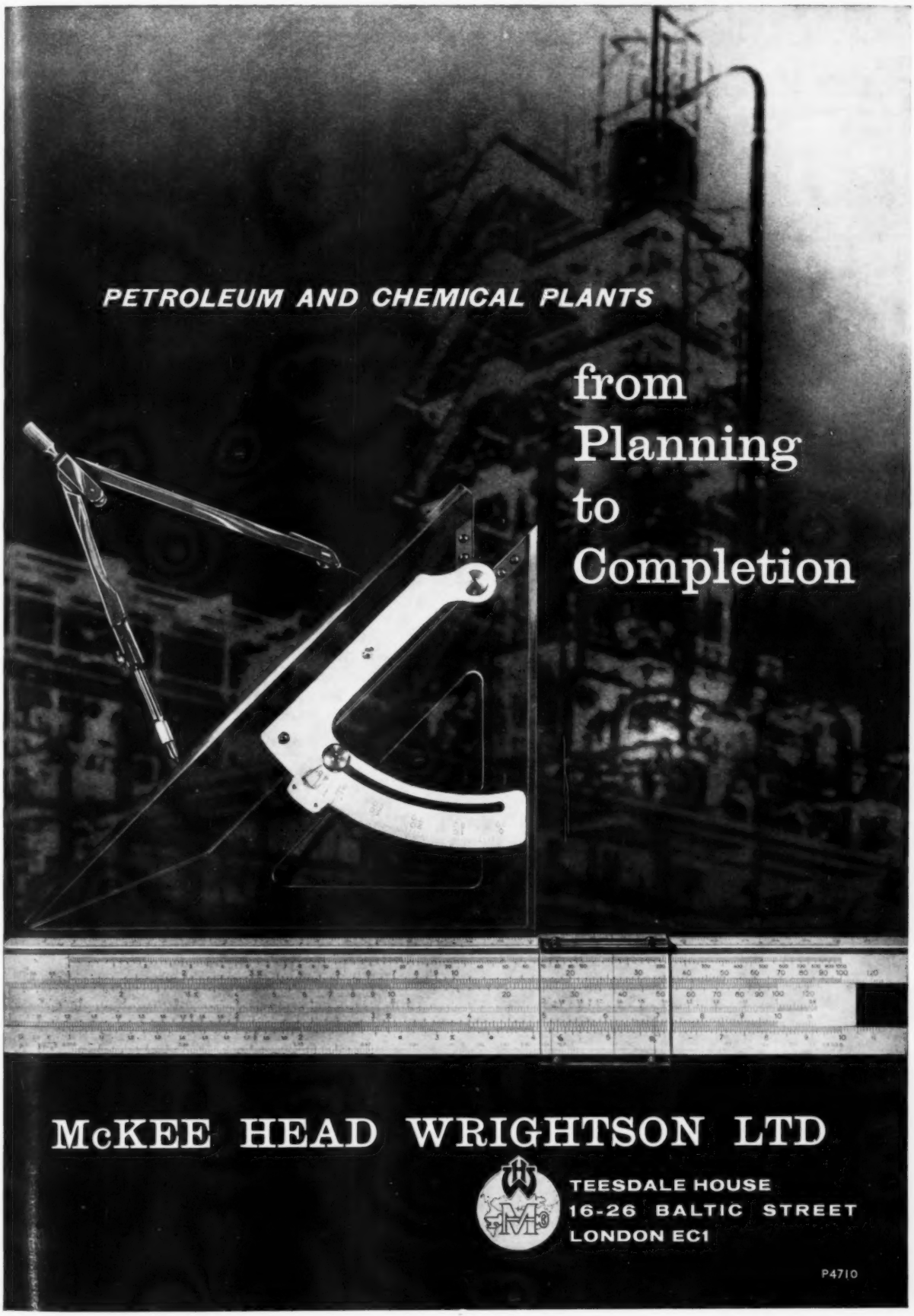
THE effect of vibration and pulsation upon convective heat transfer appears first to have been investigated successfully some years ago by MARTINELLI and BOELTER who succeeded in obtaining a five-fold increase in heat transfer coefficient when they subjected a $\frac{3}{4}$ in. diameter tube to vertical sinusoidal vibrations at frequencies up to 40 cycles per second. Of other investigators LEMLICH (now in the Department of Chemical Engineering, University of Cincinnati, Ohio) studied the effect of vertical and horizontal vibrations in air on natural convective heat transfer from small diameter cylindrical wires. He obtained up to three-fold increases in film coefficient, and applied frequencies in the range 39-122 cps. LEMLICH suggested the vibration "stretching" of the film to explain the increased heat transfer coefficient. Other investigators also confirmed the increase in coefficient for the conditions for other experiments although the magnitudes of the increase varied considerably.

LEMLICH, working with LEVY (of The Experiment Station E.I. du Pont de Nemours & Co., Wilmington, Delaware), has since concerned himself with the effect of vibrations upon mass transfer rates from the surface of wires coated with naphthalene or d-camphor which were subsequently subjected to sinusoidal vibrations in the frequency range 20-118 cps. By and large the results obtained were qualitatively in accord with the results of the earlier heat transfer investigations. The vibrating surface produced considerable increases in natural convective mass transfer, the coefficient increasing by more than six-fold; it increases with both amplitude and frequency, the influence of the former being the more pronounced. The earlier "stretched" film concept has been used once more by LEMLICH and the effect of the vibrational variables has been correlated in terms of a "stretched" vibrational Reynolds number.

Treating Slurry from Blast Furnace Gas Cleaning Plants

IN blast furnaces, a good deal of iron is lost in the form of fine flue dust carried off with the gases. After separation in the gas cleaning plant, this fine dust is usually dumped on the spoil heaps. In Czechoslovakia,* it has been found that wet electromagnetic separation affords a reasonable economic method of reclaiming fine flue dust.

The fine dust particles carried by the gas undergo magnetic roasting in the upper part of the shaft, the off-takes and the gas mains, and are therefore susceptible to separation by a magnetic field. On the way to the spoil dump the slurry from the gas cleaning plant, thickened in a Dorr thickener, is passed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SPECIAL REVIEW ISSUE

The Fifth Anniversary of the founding of *British Chemical Engineering* will be marked by the May, 1961, number which will be a Special Review Issue including articles on:

Adsorption Processes
by J. W. CARTER,

Mass Transfer
by G. V. JEFFREYS,

Applied Reaction Kinetics
by L. K. DORAISWAMY,

Fluidization
by J. S. M. BOTTERILL,

Stirred Tanks and Mixers
by R. E. TREYBAL.

through a belt-type wet electromagnetic separator. A layer of quicklime previously deposited on the belt prevents the concentrate sticking to the belt and absorbs excess water. Raw slurry containing 18-26 per cent Fe yielded a concentrate with 52-62 per cent Fe and 5-7 per cent SiO_2. The recovery of iron varied between 60 and 70 per cent.

One electromagnetic separator with 1000 mm wide belt can process in a year about 300,000 cu m of slurry containing about 75,000 tons of flue dust—roughly the quantity discharged by four 1000 cu m blast furnaces.

The production costs per ton of concentrate are (40Cz crowns are equivalent to £1 sterling):

Wages, maintenance, amortisation, power and overhead	9.5	Cz crowns
Quicklime	12.3	„
Total	21.8	Cz crowns

* KAREL NIEMIEC, Technical Digest, December 1960, 43.

UDMH—High Energy Rocket Propellent

CONSIDERABLE attention is being devoted to the chemical engineering of both solid and liquid propellent chemicals, a case in point being unsymmetrical hydrazine. The high performance, excellent storability and ease of handling of the compound have made it increasingly popular as a liquid propellent, and it has been used as fuel with fuming nitric acid or nitrogen tetroxide oxidant, or in conjunction with other fuels to improve combustion characteristics. Some engines, which have the ability to restart in space, use 100 per cent UDMH as fuel with fuming nitric acid. Hence cryogenic propellents are being replaced by storable propellents incorporating UDMH.

The two most important manufacturing processes are: (1) a modification of the Raschig hydrazine process, and (2) nitrosation of dimethylamine to N-nitrosodimethylamine and subsequent reduction to UDMH. According to L. H. DIAMOND* the Raschig process has the disadvantage of low concentration of the product in the synthesis liquor—only 2 to 3 per cent by weight of UDMH, the balance being salt and water; and the separation of UDMH from dilute aqueous solutions is extremely difficult.

Addition of sodium hydroxide markedly improves the separation, but it is usually more efficient to use several columns rather than a single column.

Besides reviewing the chemical engineering aspects of UDMH manufacture, which include equilibrium data necessary for the purification of nitrosodimethylamine and UDMH DIAMOND summarises long term experiences of the storing and handling of the propellent, and provides guidance on materials of construction.

For processing plant and storage vessels mild steel has proved suitable. However, the unique solvent properties of UDMH limit the non-metallic materials with which it can be used although such rigid plastics as PTFE, nylon and polyethylene are suitably resistant.

* L. H. DIAMOND: "The Manufacture of Unsymmetrical Dimethylhydrazine." 53rd Annual General Meeting of the A.I.Ch.E., December 1960.

DIARY

Society of Chemical Industry

April 7. "Present Trends of Developments in Chemicals from Petroleum in France", by MONS. P. A. LAURENT at **6 p.m.** 14 Belgrave Square, London, S.W.1.

April 27. Annual General Meeting. "Recent Microbiological Problems in Industry", by H. J. BUNKER at **6 p.m.** 14 Belgrave Square, S.W.1.

The Society of Instrument Technology

April 25. "Instrumentation in the Iron and Steel Industry", by A. H. POPLE. Manson House, 26 Portland Place, London, W.1.

The 1st International Congress on Metallic Corrosion

April 10-15. Imperial College of Science & Technology, South Kensington, London, S.W.7.

The Institution of Chemical Engineers

April 12. Chemical Engineering Dept., The University of Birmingham, Edgbaston, Birmingham 15, **9.30 a.m. to 5.30 p.m.** Symposium.

Morning Session: New metals and alloys as materials of construction.

Afternoon Session: New non-metallic materials of construction.

April 18. North Western Branch. Venue to be arranged. Joint meeting with the Institute of Petroleum. "Recent developments in the production of olefines and aromatics by petroleum cracking" by M. RUHEMANN (Member), D. R. CUMMINGS (Graduate) and W. L. SEDDON (Associate Member).

April 19. North Western Branch, Grosvenor Museum, Chester at **7.30 p.m.** "Some corrosion problems in the chemical industry" by D. BENNION (Graduate).

April 25. The Park Lane Hotel, Piccadilly, London, W.1. 39th Annual General Meeting and Annual Dinner and Dance.

Institute of Welding Spring Meeting 1961

April 28. So as to coincide with the Engineering, Marine, Welding and Nuclear Energy Exhibition at Olympia.

April 25-28. Recent Developments in the Welding and Allied Processes.

The Institute of Refrigeration

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Metallurgical Society of the A.I.M.E.

April 12-14. International Symposium on Agglomeration. 29, West 39th Street, New York 18, N.Y.

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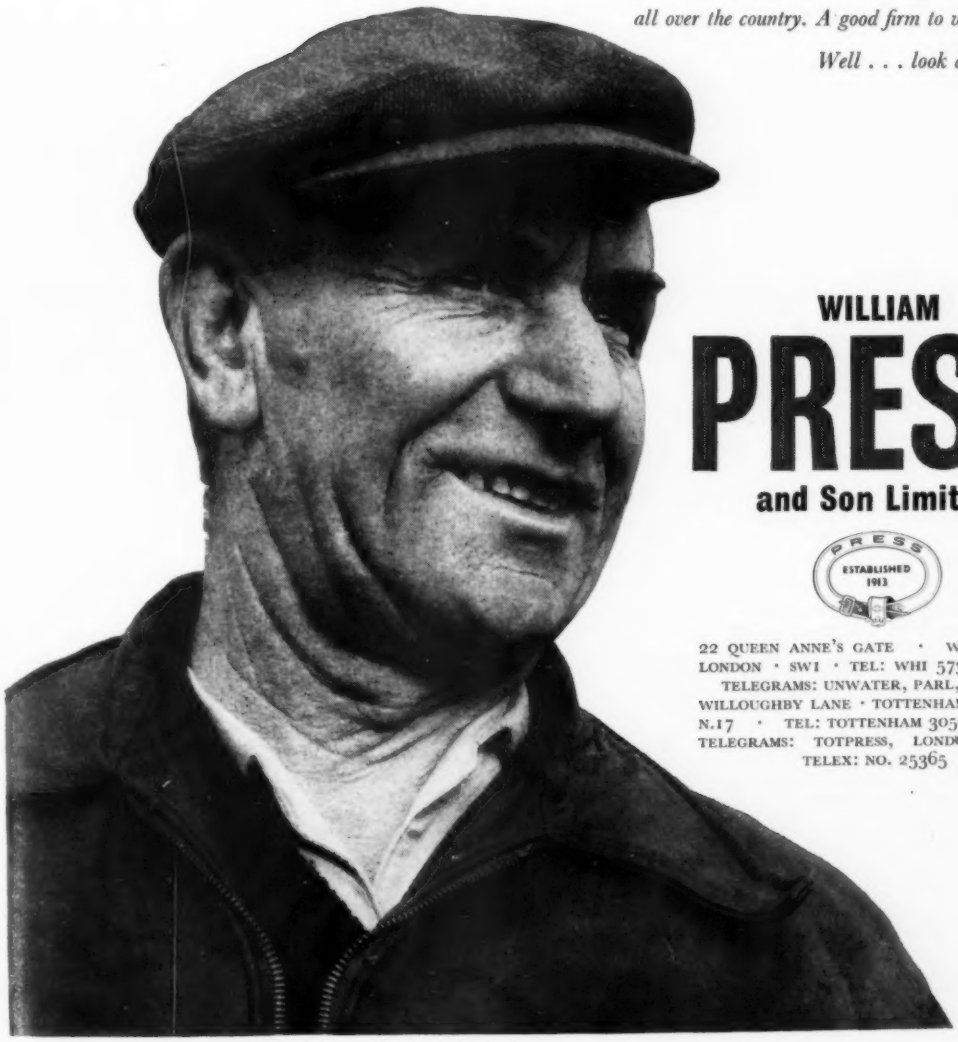

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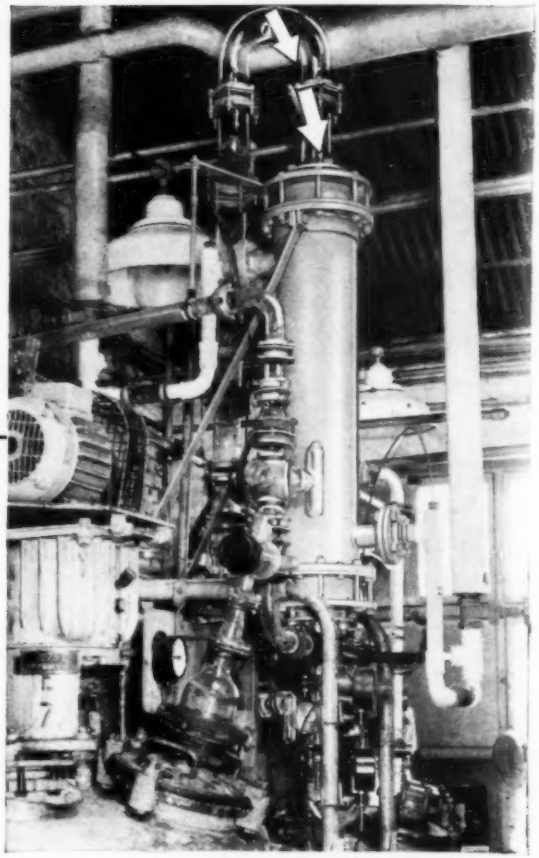

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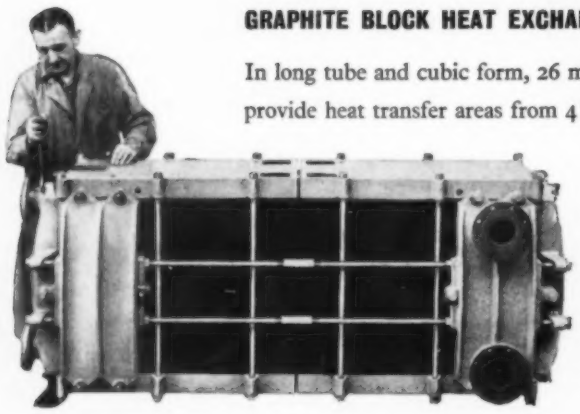

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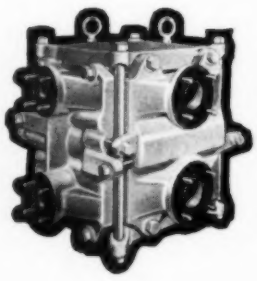

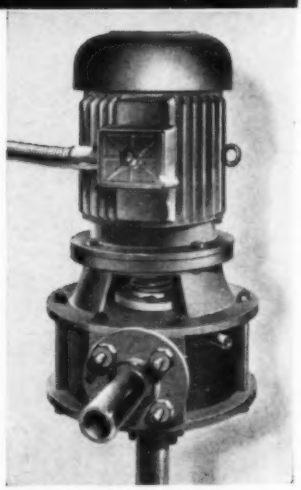

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A Uniform Code Needed

ONE need only refer to a few of the processes carried out at pressure, such as the synthesis of ammonia, methyl alcohol, urea, the various hydrogenation processes and the manufacture of petrochemicals, to appreciate the importance of pressure vessels to the processing industries. Apart from their use, another vital aspect of pressure vessels is their mechanical design. At present the equipment of this kind is made in various parts of the world according to systems of rules which lay down standards for materials of construction, plate thicknesses, procedures for testing and devices which must be installed to ensure safe operation.

These various codes owe their existence mainly to the need for safety, a need which became apparent in the early part of the nineteenth century after the development of Watt's steam engine, which created a demand for steam at much higher pressures than had been required by earlier devices. However, the higher pressures required led to many boiler explosions and accompanying fatalities, a particularly disastrous event causing a Parliamentary Committee to be formed, charged with the task of finding the causes. This committee concluded that the responsible factors were unsuitable materials, improper construction and excessive pressure, and they urged that future vessels should employ hemispherical or segmental heads for cylindrical shells, wrought iron should be the constructional material and safety valves should be mandatory on all steam-raising equipment.

With the wealth of knowledge and experience that has been gained since those early days, it is easy to see that these recommendations could not suffice, and such was the serious public concern at the loss of life and damage to property arising from numerous pressure vessel failures, which continued to occur right up to the middle of the last century, that effective prevention measures became absolutely necessary. It is perhaps significant that this was at a period when FRANKLAND required autoclaves for his processes and when W. H. PERKIN Senior's discovery of synthetic dyes led to a rapidly expanding demand for large pressure vessels.

To meet the needs of the day a body of plant owners was formed which offered to inspect pressure equipment periodically and give advice on its construction, maintenance and safe working. From this body sprang the engineering insurance companies, which offered a cover to users of high-pressure equipment against loss of life and damage to property. This service required strict adherence to the rules of construction, design, material specifications and operation, which these companies formulated.

Today the direct descendants of these rules, the various codes governing the mechanical design of pressure vessels, have succeeded in ensuring a high level of safety; apart from this, they have other valuable functions to perform, in particular in the realm of standardisation. Both the user and maker stand to gain a great deal by a greater degree of standardisation that at present prevails, for standardisation can reduce costs at all levels. For example, at the design level by simplifying design procedures, and at the supply level by standardising on the size and design of various details of vessels such as nozzles, ends, manholes and so on.

In Britain the main obstacle to effective standardisation is the existence of too many codes. There are at least three codes which apply to pressure vessels; namely, the British Standard codes and the rules of Lloyd's and the A.O.T.C. In addition, the A.S.M.E. and A.S.M.E.-A.P.I. codes are widely used and at least one user firm has its own standards. Another difficulty arising partly at least from the multiplicity of British codes is the existence of many specifications for constructional materials for pressure vessels, and there are even standards which compete in this sphere. The trouble here is that there will be a tendency to use the material of inferior quality; if more than one quality level is required, then conditions of the use for each grade should be accurately defined.

Apart from the numerous material specifications the existence of several codes results in varying standards being set for vessels for similar duties. This state of affairs multiplies the amount of drawing office work and retards standardisation of construction.

Codes also have an educational role to play, since they disseminate the accumulated experience acquired by engineers in industry and are, therefore, essential vehicles of instruction. But their successfulness in this direction depends upon their quality and the degree to which they give straightforward answers to questions arising daily in industry. The existence of several codes only leads to confusion and deprives any of them of a truly authoritative status.

A number of other disadvantages arise from the existence of several codes; one is the lack of a single body charged with their administration. As a result, a uniform interpretation of cases not covered by available codes is not possible, a respect in which other countries are more fortunate. Another is that vexed question of the allowable design stress; first, whether it should be increased, and, secondly, whether its basis should be changed to the yield stress. It can well be argued that the latter would make the design process more rational and vessels cheaper.

While the difficulties of code unification are not overestimated, the benefits which the step would provide are virtually beyond dispute. It is also well to keep our eye on the international scene. The appearance of the I.S.O. code, which may now be nearing the concluding stages of formulation, and which, incidentally, uses the yield point as the design stress criterion, is likely to stimulate the demand in this country for a single code.

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GAS DISPERSION IN SOLID-LIQUID FLUIDISED BEDS

An account of a photographic study on the gas dispersion in solid-liquid fluidised beds. The effect of the bed properties, including such variables as height, diameter and density of the particles, void degree, has been considered, and ascent velocities of gas bubbles in solid-liquid fluidised beds have been measured. A model is developed which interprets some aspects of the behaviour of solid-liquid-gas fluidised systems and provides a basis for calculation of interfacial area between gas and solid-liquid suspension in such systems

by L. MASSIMILLA, A. SOLIMANDO and E. SQUILLACE

THE investigation described in this article is part of a research on solid-liquid-gas systems obtained by injecting gas in beds of solid particles fluidised with liquids.

Previous experiments[1] on CO_2 absorption in water, with or without fluidised particles, have shown that, at constant gas and liquid space velocities, the transfer rates are higher in the second than in the first case. This fact has been explained by assuming that particles reduce the specific contact surface between the gas and the liquid. Particles would affect the specific contact surface by both (1) increasing the average diameter of the bubbles formed immediately above the nozzle, or the porous plate, as the dispersion occurs and (2) promoting their coalescence while rising in the bed.

The present investigation aims to isolate and study separately these two phenomena, by determining the average diameters of bubbles emerging from fluidised beds of different heights. Bed expansion, nature and average diameter of the particles, flow rate of the injected gas and nozzle diameter are considered, as far as their effects on the gas dispersion are concerned. In addition, data are given on the rise velocities of bubbles in solid-liquid fluidised systems, which are important in studying the above-mentioned phenomenon of the bubble coalescence.

Experimental

The solid-liquid fluidised systems used in this investigation were obtained by fluidising by means of water beds of particles made of silica sand ($D_s = 0.0086$ in.), glass beads "A" ($D_s = 0.031$ in.), glass beads "B" ($D_s = 0.043$ in.) and iron sand ($D_s = 0.0102$ in.). The water used was derived directly from the town domestic supply at a temperature of about 60°F. The gas dispersed was air. It was injected in the solid-liquid systems by means of nozzles 2.4 in. long, made of stainless-steel tubes 0.04 in. and 0.015 in. I.D. Fig. 1 shows the apparatus used in a photographic study of the gas dispersion in solid-liquid fluidised beds. The fluidised column (*a*) was 4 ft long with a rectangular cross-section of 3.4 in. × 2.4 in. This column was made of glass sheet and was connected at the top to a disengaging section (*b*) and at its base to a calming section (*c*). A screen plate was placed between the column (*a*) and the section (*c*). On the bottom of (*c*) two tubular connections (*d*) were used as water inlets. A third connector (*e*) was connected to nozzle (*f*). The tubes (d) were connected to a water pump. The water flow rate was controlled by means of a gate valve (*h*) and measured by means of a flowmeter (*g*). The nozzle (*f*) was connected to a device for the measurement of the injected gas flow rate. It was composed of a graduated tank of 2 gal. (*i*), with a manometer (*l*), a head column of 1 in. I.D. (*m*), a water tank (*n*), which was kept at a constant level by means of a spill way (*o*), a nozzle (*p*) feeding the head column (*m*).

Fig. 2 shows the apparatus used for measuring the rise velocities of bubbles in solid-liquid fluidised systems. The fluidising column (*a*) was made of a 3.5 in. I.D. tube of a transparent synthetic material (Perspex). It was 5.2 ft long and terminated at its upper end with a disengaging section (*b*). Between the column (*a*) and the calming section (*c*) was interposed porous plate (*d*). A device for the injection of single bubbles in the column was placed 4.3 in. from the porous plate. This device was formed of a glass injection tube (*e*), a glass inspection tube (*f*) and a rubber tube (*g*). Injection tubes of 0.09-0.12 and 0.17 in. I.D. were used. A "T" piece was placed between the inspection tube (*f*) and the rubber tube (*g*) and connected to a water tank (*l*) 13 ft from the ground level. A stopcock (*m*) connected the tank (*l*) to the injection device. The water flow rate was controlled by means of the gate valve (*n*) and measured by means of the flowmeter (*o*).

The following experimental technique was used in the photographic study carried out by means of the apparatus of Fig. 1. The solid particles were poured in the column (*a*) and carefully packed. The height of the bed was measured and the particles fluidised. The tank (*i*) was then pressurised by admitting water from the tank (*n*) to the

L. MASSIMILLA, a Chemical Engineering graduate of Naples University, has worked on fluidisation for several years and is now a lecturer in Chemical Engineering at Naples University. A. SOLIMANDO, also a graduate of Naples University, has joined the Società "Montecatini" and is now working at Porto-Empedocle, Agrigento, Italy. E. SQUILLACE graduated in Naples and has since joined the Società "Edison" and is now working in Milan.

Symbols Used

a, b = numerical values, dimensionless;
C', C'', C''', C^{**}, C^{*}, C = fractions of passes which result in collisions with respect to the total number of passes, dimensionless;
d_n = nozzle diameter, in.;
D_b = diameter of a single bubble, in.;
$D_{m'}$ = average diameter of the bubbles for a given experimental condition, in.;
D_{mzz} = average diameter of the bubbles at a distance Z from the nozzle, in.;
$D_{mZ/z'}$ = average diameter of the bubbles at a distance $Z' = Z + \triangle Z$ from the nozzle, in.;
D_{mo} = average diameter of the bubbles above the nozzle, in.;
D_p, D_m, D_g = average diameter of the bubbles forming the groups of small, medium or large bubbles, in.;
D_{ps}, D_{ms}, D_{gs} = average diameter of the cross-section of the average small, medium or large bubble normal to the flow direction, in.;
D_s = average equivalent diameter of the particles, in.;
G = gas flow rate, cfm^{-1};
L_o = height of the packed bed of particles, ft;
L = height of the fluidised bed of particles, ft;
$\triangle L = L - L_o$ = expansion of the fluidised bed of particles, ft;
$L_o/\triangle L$ = reciprocal of the expansion degree of the fluidised bed, dimensionless;
n_p, n_m, n_g = number of the small, medium or large bubbles flowing through the section ZZ per unit time, sec^{-1};
$\triangle N^{*}$ = number of passes occurring per unit time between ZZ and $Z'Z'$, sec^{-1};
$\triangle N$ = number of collisions occurring per unit time between ZZ and $Z'Z'$, sec^{-1};
S = cross-section of the vessel containing the fluidised bed, $in.^2$;
S' = cross-section of the path through which bubbles flow, $in.^2$;
V_p, V_m, V_g = average rise velocity of a single small, medium or large bubble, ft sec^{-1};
V_b = rise velocity of a single bubble, ft sec^{-1};
Z = distance from the nozzle, ft.

TABLE I—Glass Beads "A"
Reciprocal of the expansion degree of the fluidised bed: $L_o / \triangle L = 1.9$

Nozzle diameter d_n, in.	Gas flow rate G, cfm^{-1}	Average bubble diameter D_{mo}, in.
0.015	0.0011	0.125
0.015	0.0028	0.149
0.015	0.0053	0.165
0.040	0.0053	0.189
0.040	0.0095	0.189
0.040	0.0138	0.204

head column (*m*). As soon as the water reached a sufficient level in the head column, air began to efflux from the nozzle. At steady-state conditions: (1) the water flow rate, controlled by means of the valve (*h*), was sufficient to ensure the desired bed expansion; (2) the pressure in the tank (*i*), measured by means of the manometer (*l*), was constant; (3) the gas flow rate injected in the column (*a*) was practically equal to the flow rate of water leaving the nozzle (*p*). Considering that the head in the tank (*n*) was maintained constant by means of the spill way (*o*), different flow rates of water (and of air) were obtained by changing the efflux section of the nozzle (*p*).

The gas flow rate injected in the column was directly measured by determining the displacement of the water level in the graduate tank (*i*) in the interval of time in which the run was carried out. By means of this technique it was possible to obtain that the gas flow rate remained constant throughout each run, even in the case of the smallest flow rates used (maximum deviation, 3 per cent).

As soon as the steady-state conditions were reached, stills were taken of the part of the column (*a*) immediately above the fluidised bed. A Retinette IIA Kodak camera was used with an Agfa Isopan negative film. The field taken was of about 5.5 in. × 8.3 in. It was illuminated by a Braun electronic flash. For each working condition a minimum of 8 and a maximum of 20 still pictures were taken. Negative films were developed and read by means of a projector.

The diameter of each bubble emerging from the fluidised bed was determined by averaging its longest and shortest diameter. For a given experimental condition, the average diameter of the bubbles was obtained as an average of the diameters of all the bubbles appearing in all the pictures taken. Average diameters were determined on the basis of a maximum of 250 and a minimum of about 50 bubbles.

The apparatus of Fig. 2 was operated as follows:

First, the fluidised bed was obtained by regulating the gate valve (*n*) and a bubble was introduced in the inspection tube (*f*) by squeezing the rubber tube (*g*) and operating the stopcock (*m*). During this operation, the pinchcock (*h*) was open and the pinchcock (*i*) was closed. The bubble was then admitted to the injection tube (*e*), where it either remained as a whole or was fractionated depending on its magnitude, the size of the tube and the opening of the stopcock (*m*). One bubble at a time was isolated in the injection tube (*e*), measured and injected by operating the stopcock (*m*). A stopwatch was used to measure the interval of time between the instant of the injection and the instant at which the bubble emerged from the fluidised bed. The length basis for bubble velocity measurement was 4.5 ft. When necessary, the pinchcock (*i*) was opened to renew the air supply in the tube (*g*).

Results

Figs 3 to 9 and Table I show the effect of the variables considered in this investigation on gas dispersion in solid-liquid fluidised beds.

Fig. 3 and Table I show the effect of the particle diameter, bed expansion, gas flow rate and nozzle diameter on formation of gas bubbles in solid-liquid fluidised beds. The average diameter, D_{mo}, in both Fig. 3 and Table I is referred to experiments with shallow beds. In these experiments the distance between the top of the bed and the nozzle orifice was about 2 in. Because of this short distance, it has been assumed that the effect of the bubble coalescence could be disregarded.

The effect of the coalescence is outlined in Figs. 4 to 9 for different values of the nozzle diameter and gas flow rates and for several solid-liquid fluidised beds. Following the above-mentioned experimental procedure, the average diameters, D_{mzz}, have been obtained on the basis of the diameters of the bubbles emerging from the top of the beds at distances Z from the nozzle. Therefore, in plotting data in Figs. 4 to 9, it has been implicitly assumed that D_{mzz} does not depend on the height of the bed above the level Z, whatever this height may be.

It has been also supposed that the increase of D_{mzz} with Z depends only on bubble coalescence. The effect of reduction of pressure on the size of the bubbles, while rising in the bed, has been disregarded.

In Fig. 10 the values of the specific contact surfaces between the gas and the solid-liquid system are given as a function of the average diameter, D'_m. For a given experimental condition, these values have been determined by

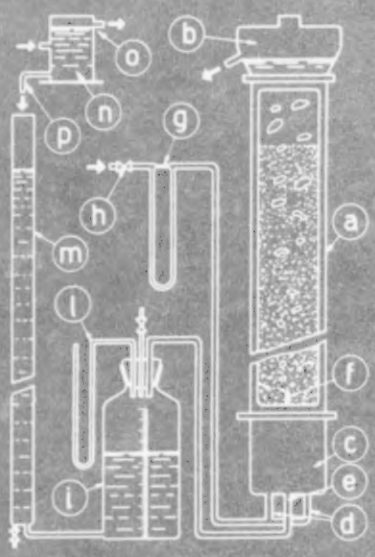

Fig. 1

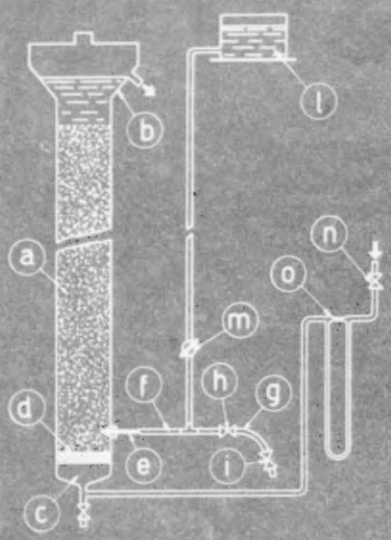

Fig. 2.

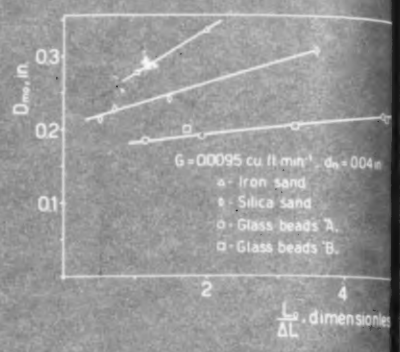

Fig. 3.

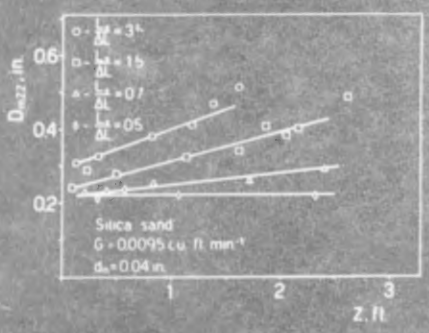

Fig. 4.

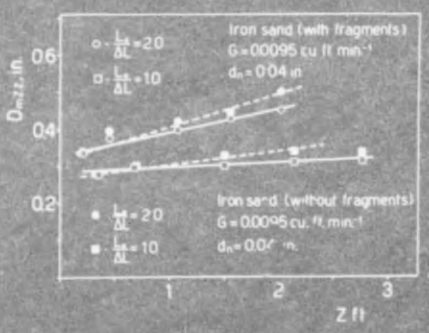

Fig. 5.

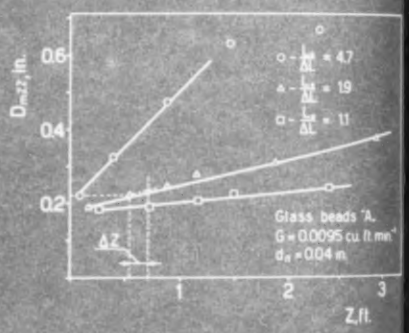

Fig. 6.

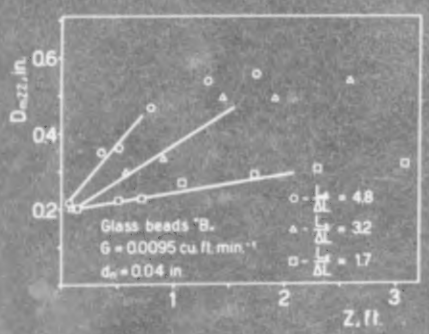

Fig. 7.

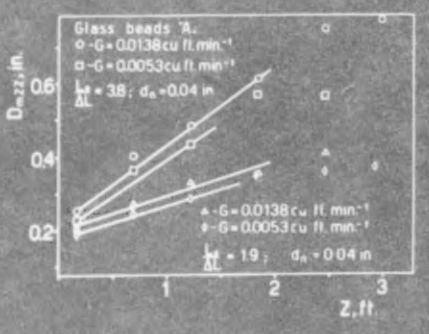

Fig. 8.

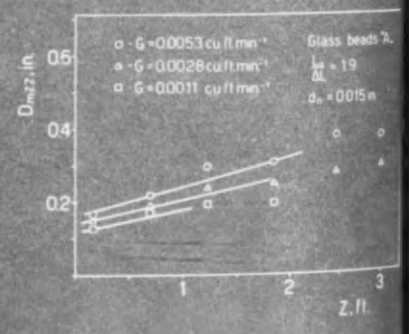

Fig. 9.

Fig. 1. Apparatus used in photographic study of gas dispersion.

Fig. 2. Apparatus used for determination of bubble ascent velocities.

Fig. 3. The effect of particle density and diameter and bed expansion on gas bubble formation.

Figs. 4-9. The effect of coalescence for different values of nozzle diameter and gas flow rates for several solid-liquid fluidized beds.

Fig. 10. Specific contact surface between gas and solid-liquid system as a function of average bubble diameter.

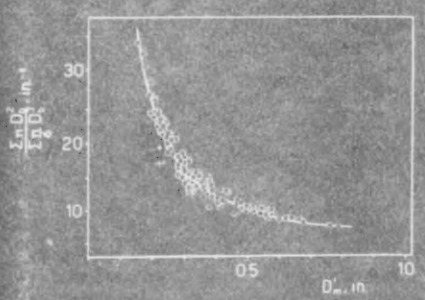

Fig. 10.

dividing the surface summation $\Sigma\pi D_b^2$ by the volume summation $\Sigma\frac{\pi}{6}D_b^3$ extended to all the bubbles pictured.

Finally, diagrams, rise velocity vs. bubble diameter, are plotted in Figs. 11 to 16. These data have been obtained by means of the apparatus shown in Fig. 2.

The effect of the particles on the formation of gas bubbles in solid-liquid fluidised beds is rather complex. Both the properties of the bed and the characteristics of the gas injection affect the gas dispersion immediately above the nozzle, as it is shown in Fig. 3 and Table I. A comparison between the gas dispersion in liquids and solid-liquid fluidised systems would seem a good approach to the theoretical study of the phenomenon. However, our attempt to interpret the experimental data, on the basis of the correlations valid in the case of the formation of the gas bubbles in liquids, met with little success.

On the contrary, the effect of the particles on the coalescence of the gas bubbles rising in solid-liquid fluidised beds seems to be easier to study. Figs. 4 to 9 show that the trend of the diagrams, bubble average diameter vs. distance from the nozzle, depends slightly on the nature and diameter of the particles, as well as on the nozzle diameter and gas flow rate. It depends mainly on bed expansion.

Besides, as a first approximation, it may be assumed that the average bubble diameters increase proportionately with the distances from the nozzle, at least up to certain values of such distances. Therefore, the following correlation may be written:

$$\frac{D_{mzz} - D_{mo}}{Z} = a\left(\frac{L_o}{\triangle L}\right)^b \qquad \ldots.(1)$$

in which D_{mzz} is the average diameter at a distance Z from the nozzle; D_{mo} is the average bubble diameter immediately above the nozzle; $L_o/\triangle L$ is the reciprocal of the degree of the bed expansion; a and b are numerical coefficients. Of course, Equation (1) may be of interest only in the range of Z in which proportionality between Z and D_{mzz} exists. Figs. 4 to 9 show that this range itself depends on bed expansion, increasing as the bed expansion increases.

By means of the diagram of Fig. 17 the values of a and b in Equation (1) have been obtained on the basis of the data of all the experiments carried out. Values of 2.7×10^{-3} and of 1.3 have resulted for a and b respectively. Dispersion of data in diagram of Fig. 17 is not surprising. Equation (1) has no physical meaning and is empirical. A more fundamental study of the diagrams, bubble average diameter vs. distance from the nozzle, will be carried out in the following part of this article.

Coalescence of Bubbles in Solid-liquid Fluidised Systems

The coalescence of bubbles rising in solid-liquid fluidised systems may be investigated considering the bubble flow pattern in such systems, namely, the dependence of the rise velocity on the bubble diameter. Two bubbles leaving the nozzle at different times can indeed collide only in the case of their rise velocities being different. The slight tendency of bubbles to coalesce while rising in vessels containing water depends on the fact that the rise velocity of bubbles in water is practically constant in a considerable range of the bubble diameter (from 0.12-0.78 in.). As it is shown in Fig. 18, rise velocity of bubbles in water[2] increases rapidly as the diameter increases. It reaches a maximum at about 0.06 in., it decreases slowly between 0.06 and 0.27 in., and then, still slowly, it increases again. The curves, velocity of ascent vs. bubble diameter for solid-liquid fluidised systems, follow a completely different pattern.

These curves compare approximately with the analogous curves which may be found for viscous liquids, as shown in Fig. 18.[3]

In Fig. 18 the experimental curve for a solid-liquid fluidised bed has been extended to small bubble diameters using the following equations by STOKES:

$$V_b = \frac{2}{9}\frac{\rho}{\mu} g r^2; \quad 0 < \frac{V_b r \rho}{\mu} \leq 0.3 \qquad \ldots.(2)$$

and OSEEN[4]:

$$V_b = -\frac{1.33\mu}{r\rho} + \sqrt{\left(\frac{1.33\mu}{\rho r}\right)^2 + 0.593 g r} \; ; \; 0.3 < \frac{V_b r \rho}{\mu} \leq 1 \qquad \ldots.(2')$$

In these equations V_b is the bubble velocity; r its radius; μ and ρ are the viscosity and the density of the medium in which the bubble is moving; g is the gravity constant.

A good extrapolation has been obtained by assuming μ equal to 62.5 cp. It is interesting to note that this value is of the same order of magnitude as those which have been found by TRAWINSKI[5] for fluidised beds similar to that considered.

Contrarily to what happens in the case of water, for both a viscous liquid and a solid-liquid fluidised system, the rise velocity increases quite steadily as the bubble diameter increases. As a consequence, there is a large variation in ascent velocities with the bubble diameter.

Figs. 11 to 16 show that the shape of the curves, rise velocity vs. bubble diameter, is practically determined by the bed expansion, whatever may be the nature and diameter of the particles forming the fluidised bed. This circumstance explains how it has been possible to correlate the bubble average diameter with the distance from the nozzle by means of Equation (1). According to such equation, the slope of the straight line, bubble velocity vs. distance from the nozzle, is a function of the bed expansion, and thus it accounts, although empirically, for the influence of this parameter on the rise velocity of the bubbles.

Considering the curves, rise velocity vs. bubble diameter, it may be explained, at least qualitatively, why in some diagrams of Figs. 4 to 9 the average bubble diameter levels off when the distance from the nozzle increases. As coalescence progresses, the diameters of the bubbles increase. Then, because of the shape of the curves of Figs. 11 to 16, the differences between the velocities of the bubbles rising in the bed decrease; consequently, the possibility of the bubbles colliding also decreases.

It should be pointed out, however, that the variation of the rise velocity with the bubble diameter cannot completely explain the trend of the diagrams of Figs. 4 to 9. Other effects must be considered, which depend on the gas flow rate and the nozzle diameter, in addition to the bed expansion.

A quantitative analysis of the effect of the shape of the curve, rise velocity vs. bubble diameter, on the bubble coalescence is complicated because of the large variety of diameters of the bubbles produced by the disruption of the gas jet flowing through the nozzle. Each bubble rises in the fluidised bed with a velocity depending mainly on its diameter; it may pass smaller bubbles and be passed by larger ones. Therefore, it is difficult to account for the totality of passes which take place at a given level of the fluidised bed and for the fraction of them which resolve in collisions between bubbles.

As a simpification, one may accept the division of the bubbles in groups according to their diameters, and assume for each one a diameter which is the average of the diameters of all the bubbles belonging to the group.

Let us divide the bubbles into two groups, those of large and small bubbles, and indicate as D_g, V_g, D_p and V_p, respectively, the average diameters and velocities of the groups of large and small bubbles. Let us also indicate as n_g and n_p the numbers of large and small bubbles flowing per unit time through an arbitrary section ZZ of a solid-liquid fluidised bed at a distance Z from the nozzle, $Z'Z'$ being another section at a distance $Z + \Delta Z$ from the nozzle, let us determine the number of passes which take place per unit time between ZZ and $Z'Z'$.

The average small bubble covers the distance ΔZ in a time Δt_p given by:

$$\Delta t_p = \frac{\Delta Z}{V_p} \qquad \ldots.(3)$$

Assuming that this bubble flow through ZZ at a time $t = 0$, it is passed by all the bubbles flowing through the same section in the interval of time $\Delta t'$, between $t = 0$ and $t = 0 + \Delta t'$ given by:

$$\Delta t_p V_p = (\Delta t_p - \Delta t') V_g$$

whence

$$\Delta t' = \frac{V_g - V_p}{V_g} \Delta t_p$$

and according to Equation (3):

$$\Delta t' = \frac{V_g - V_p}{V_g} \frac{\Delta Z}{V_p}$$

Also, if n_g is the number of large bubbles flowing through ZZ per unit time, then the relation

$$n_g \, \Delta t' = n_g \left(\frac{1}{V_p} - \frac{1}{V_g}\right) \Delta Z$$

indicates how many times each small bubble is passed by large bubbles between ZZ and $Z'Z'$. Also, n_p being the number of small bubbles flowing through ZZ per unit time, the total number of passes per unit time is:

$$\Delta N^* = n_g \, n_p \left(\frac{1}{V_p} - \frac{1}{V_g}\right) \Delta Z$$

If C is the fraction of the passes which resolve in collisions with respect to the total number of passes, the number of collisions occurring per unit time between ZZ and $Z'Z'$ is:

$$\Delta N = C \, n_g \, n_p \left(\frac{1}{V_p} - \frac{1}{V_g}\right) \Delta Z \qquad \ldots.(4)$$

More correctly, the bubbles may be divided into three groups. In this case, if D_g, V_g, n_g; D_m, V_m, n_m; D_p, V_p, n_p indicate the average diameters, the average velocities and the numbers of bubbles respectively large, medium or small, flowing per unit time, through ZZ:

$$\Delta N' = C' \, n_g \, n_m \left(\frac{1}{V_m} - \frac{1}{V_g}\right) \Delta Z \qquad \ldots.(5)$$

$$\Delta N'' = C'' \, n_g \, n_p \left(\frac{1}{V_p} - \frac{1}{V_g}\right) \Delta Z \qquad \ldots.(6)$$

$$\Delta N''' = C''' \, n_m \, n_p \left(\frac{1}{V_p} - \frac{1}{V_m}\right) \Delta Z \qquad \ldots.(7)$$

are the numbers respectively of collisions between medium and large bubbles, small and large bubbles and small and medium bubbles occurring per unit time between ZZ and $Z'Z'$.

Then assuming that $C' = C'' = C''' = C$,

$$\Delta N = C \left[n_g \, n_m \left(\frac{1}{V_m} - \frac{1}{V_g}\right) + n_g \, n_p \left(\frac{1}{V_p} - \frac{1}{V_g}\right) + n_m \, n_p \left(\frac{1}{V_p} - \frac{1}{V_m}\right) \right] \Delta Z \qquad \ldots.(7')$$

is the number of collisions occurring per unit time between ZZ and $Z'Z'$. The parameters affecting C will be extensively discussed later. Now let us see how, on the basis of Equations (5), (6) and (7), it is possible to express the increase of the average diameter of the bubbles between ZZ and $Z'Z'$.

n_m and n_p must verify the following equation:

If G is the gas flow rate inlet at ZZ, G, D_g, D_m, D_p, n_g,

$$G = \frac{\pi}{6}\left[n_g\, D_g{}^3 + n_m\, D_m{}^3 + n_p\, D_p{}^3\right] \quad \ldots.(8)$$

According to Equations (5), (6) and (7), and assuming that no absorption or desorption takes place between ZZ and $Z'Z'$, at $Z'Z'$ the outlet is:

$$G = \frac{\pi}{6}\left[(n_p - \triangle N'' - \triangle N''')\, D_p{}^3 + (n_m - \triangle N')\, D_m{}^{*3} + n_g\, D_g{}^{*3}\right]$$

in which:

$$D_m{}^* = \left(\frac{\triangle N'''}{(n_m - \triangle N')}\, D_p{}^3 + D_m{}^3\right)^{\frac{1}{3}}$$

$$D_g{}^* = \left(\frac{\triangle N'\, D_m{}^3 + \triangle N''\, D_p{}^3}{n_g} + D_g{}^3\right)^{\frac{1}{3}}$$

Therefore, if at ZZ:

$$D_{mZZ} = \frac{n_p\, D_p + n_m\, D_m + n_g\, D_g}{n_p + n_m + n_g}$$

at $Z'Z'$:

$$D_{mZ'Z'} = \frac{(n_p - \triangle N'' - \triangle N''')\, D_p}{(n_p + n_m + n_g) - (\triangle N' + \triangle N'' + \triangle N''')} + \frac{(n_m - \triangle N')\left(\frac{\triangle N'''}{(n_m - \triangle N')}\, D_p{}^3 + D_m{}^3\right)^{\frac{1}{3}}}{(n_p + n_m + n_g) - (\triangle N' + \triangle N'' + \triangle N''')} + \frac{n_g\left(\frac{\triangle N'\, D_m{}^3 + \triangle N''\, D_p{}^3}{n_g} + D_g{}^3\right)^{\frac{1}{3}}}{(n_p + n_m + n_g) - (\triangle N' + \triangle N'' + \triangle N''')} \quad \ldots.(9)$$

Equations (5), (6), (7) and (9) may be used to calculate $C = C' = C'' = C'''$, when both diagram, average diameter vs. distance from the nozzle, and bubble size distribution are known. The calculation of a value of C is here briefly illustrated.

Example: It is desired to determine the value of C for the following experimental conditions:

Solid liquid system: glass beads "A"—water.

Distance from the nozzle: 0.533 ft.

Reciprocal of the degree of the bed expansion: 1.9.

Nozzle diameter: 0.04 in.

Gas flow rate: 0.0095 cfm^{-1}.

Twelve stills were taken in order to depict bubbles emerging from such a solid-liquid-gas fluidised system. A number of 162 bubbles has appeared on the pictures with diameters ranging from a minimum of 0.14 in. to a maximum of 0.39 in. and an average diameter of 0.222 in.

Dividing these bubbles into three groups, we have 56 bubbles with diameters between 0.14 and 0.19 in.; 54 bubbles between 0.19 and 0.26 in.; 52 bubbles between 0.26 and 0.39 in. According to this distribution, it is $D_p = 0.16$ in., $D_m = 0.22$ in. and $D_g = 0.29$ in.

Assuming that the fractions

$$\frac{n_p}{n_g + n_m + n_p}\ ;\ \frac{n_m}{n_g + n_m + n_p}\ ;\ \frac{n_g}{n_g + n_m + n_p}$$

are equal to the fractions of small, medium and large bubbles with respect to the total number of bubbles pictured, it is:

$$\frac{n_p}{n_g + n_m + n_p} = 0.345$$

$$\frac{n_m}{n_g + n_m + n_p} = 0.334$$

$$\frac{n_g}{n_g + n_m + n_p} = 0.321$$

Two of these equations and Equation (8) form a system with three unknown values: n_p, n_m and n_g.

Substituting in this system the given values of G, D_p, D_m and D_g we have $n_p = 13.8$ sec^{-1}; $n_m = 13.4$ sec^{-1}; $n_g = 12.8$ sec^{-1}.

On the other hand, from Fig. 12 we have:

$V_p = 0.54$ ft sec^{-1}; $V_m = 0.64$ ft sec^{-1}; $V_g = 0.71$ ft sec^{-1}.

Therefore, assuming $\triangle Z = 2$ in., we obtain:

$$\begin{aligned}\triangle N' &= 4.40\,C\\ \triangle N'' &= 13.05\,C\\ \triangle N''' &= 8.91\,C\end{aligned} \quad \ldots.(10)$$

The value of C is now determined by a trial procedure. It must be selected in order that the values of $\triangle N'$, $\triangle N''$ and $\triangle N'''$, substituted in Equation (9), give a value of $D_{mZ'Z'}$ equal to that obtained by the intersection of the vertical to the abscissa $Z + \triangle Z$ and the parallel to the straight line: average diameter vs. distance from the nozzle through the point of co-ordinates ($Z = 0.533$ ft; $D_{mZZ} = 0.222$ in.).

Fig. 6 gives a value of $D_{mZ'Z'}$ equal to 0.235 in. From Equations (9) and (10) C is found to be equal to 0.20.

Following this procedure, several values of C have been calculated. These values have been given in the diagram of Fig. 19 as a function of the average bubble diameter D_m'.

The interpretation of the diagram requires a preliminary analysis of the physical meaning of C and of the parameters on which it depends.

As it has been assumed before, C is the fraction of passes which result in collision with respect to the total number of passes occurring between ZZ and $Z'Z'$. It may be affected by several parameters, such as bubble size, cross-section of the container, bubble frequency and flow condition as the pass takes place.

The effect of the first two factors is evident. After all, C is the probability that a large bubble passing a small one hits it; i.e., the probability that, at the level at which the pass occurs, the centre of the large bubble is at a distance from the centre of the small one shorter than $\frac{1}{2}(D_{ps} + D_{gs})$; D_{ps} and D_{gs} being respectively the diameters of the cross-section of the small and the large bubbles normal to the flow direction.

If S is the cross-section of the container, assuming that at the pass level only two bubbles are present, and disregarding the hydrodynamic effects due to the pass, the value of C is given by the equation:

$$C^{**} = \tfrac{1}{4}\pi\,\frac{(D_{ps} + D_{gs})^2}{S} \quad \ldots.(11')$$

This is verified, of course, in the case of the bubbles spreading throughout the entire section S of the container.

If the bubbles flow through preferential paths, for instance, through channels in the core of the bed, Equation (11′) becomes:

$$C^{*} = \tfrac{1}{4}\pi\,\frac{(D_{ps} + D_{gs})^2}{S'} \quad \ldots.(11)$$

with S' smaller than S.

The frequency of the bubbles, which by itself determines n_p, n_m, n_g and, thus, $\triangle N'$, $\triangle N''$, $\triangle N'''$, according to Equations (5), (6) and (7), affects the value of C in a complex fashion.

At high gas flow rates, as the frequency of the bubbles increases, both the numerator and the denominator in Equation (11) may increase; the first because more than two bubbles meet at the same level of the bed; the second because bubbles tend to spread throughout the whole section of the container.

At low gas flow rates, as the frequency of the bubbles decreases, the numerator of Equation (11) remains $\pi(D_{ps} + D_{gs})^2$, while the denominator decreases because bubbles take off from the nozzle in a chainlike manner, one after the other.

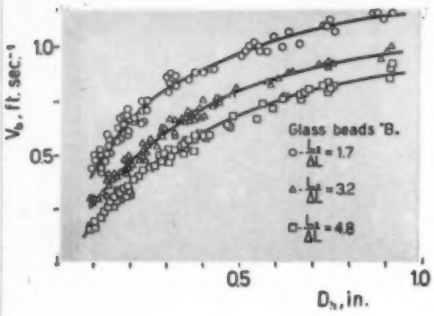

Fig. 11.

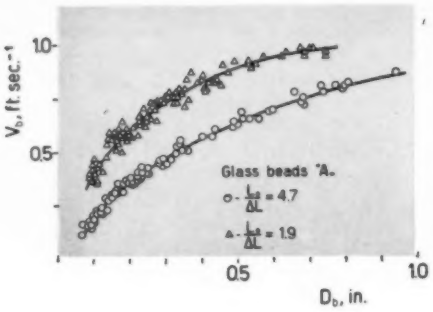

Fig. 12.

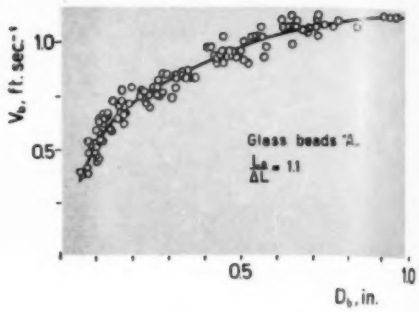

Fig. 13.

The hydrodynamic effects due to the pass have been considered, assuming that the bubbles moving upward may be regarded as stationary obstacles around which the solid-liquid fluidised system flows downward. As a consequence, the value of C has been corrected, accounting for the probability of passage of flow lines near an obstacle. Because of the lack of more suitable correlations, expressions obtained in the case of liquids flowing near obstacles[6] have been extended to the case of solid-liquid fluidised systems. A factor 3 $\left(\frac{D_{ps}}{D_{gs}}\right)$ has been assumed, which modifies Equation (11) as follows:

$$C = \tfrac{3}{4}\pi \left(\frac{D_{ps}}{D_{gs}}\right) \frac{(D_{ps} + D_{gs})^2}{S'} \qquad \ldots.(12)$$

In Fig. 19 a curve of values of C as a function of the average bubble diameter D_m' has been given along with data obtained following the procedure in the previous example. This curve has been determined by means of the equation:

$$C = \frac{\pi}{4S'}\left[\frac{D_{ps}}{D_{gs}}(D_{ps} + D_{gs})^2 + \frac{D_{ps}}{D_{ms}}(D_{ps} + D_{ms})^2 + \frac{D_{ms}}{D_{gs}}(D_{ms} + D_{gs})^2\right] \qquad \ldots.(13)$$

which supposes that the value of C, for a given experimental condition, is the average of the values of C obtained considering the three pairs of diameters (D_{ps}, D_{ms}), (D_{ps}, D_{gs}) and (D_{ms}, D_{gs}).

Three hypotheses underlie the use of Equation (13):

(1) S' is the area of the circle inscribed in the cross-section of the container with a diameter of 2.4 in. equal to the shortest side of the cross-section of the container;

(2) at the level at which a pass occurs only two bubbles are present;

(3) the diameters of the cross-sections of the bubbles normal to the flow (D_{ps}, D_{ms} and D_{gs}) may be determined as a function of the diameters (D_p, D_m and D_g), according to the correlation obtained by ROSENBERG[2] for gas bubbles rising in pure water.

Several values of C (shown as open circles on the figure) agree with the curve obtained by means of Equation (13). The values falling far from the curve (shown as black circles) are referred to experiments with iron sand ($L_o/\Delta L = 2.0$), silica sand ($L_o/\Delta L = 3.5$) and glass beads "A" for low gas flow rates ($G = 0.0011$ cfm^{-1} and $G = 0.0028$ cfm^{-1}).

In all these experiments a conspicuous channelling of the bubbles could be expected because either the bed of particles was scarcely fluidised or the bubbles left the nozzle chainwise, one after the other.

In order to account for the channelling of the bubbles, the value of S' in Equation (13) should be reduced. Thus, a new curve can be determined which, as the broken line curve of Fig. 19, correlates the higher values of C obtained.

It is necessary, however, to point out the fact that some of these values are greater than unity. Since C is by definition the ratio between passes resulting in collisions and the

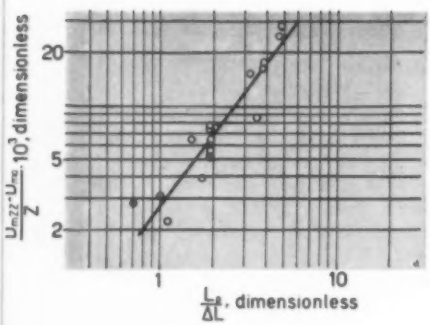

Fig. 17.

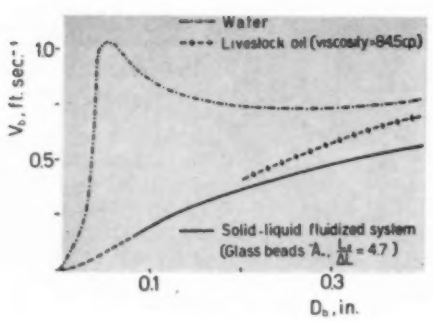

Fig. 18.

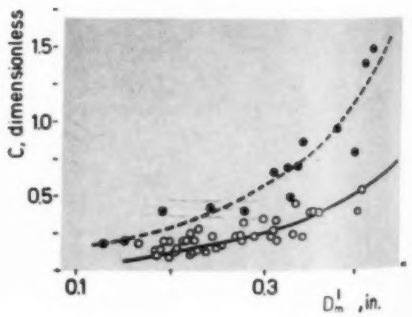

Fig. 19.

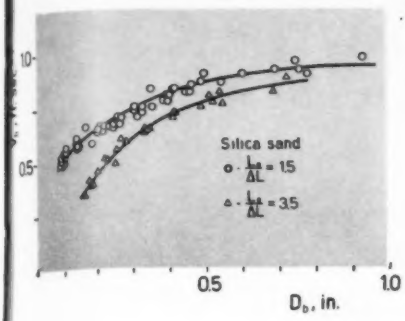

Fig. 14.

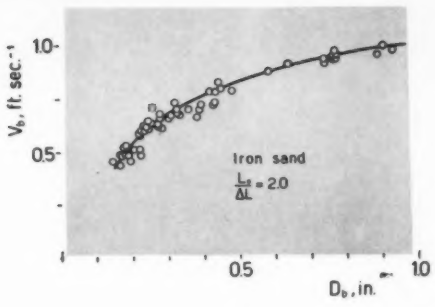

Fig. 15.

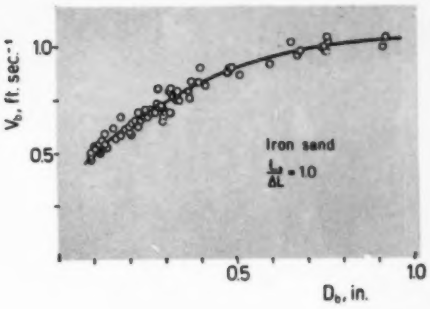

Fig. 16.

total number of passes, these values have no physical meaning. This fact outlines a weak point in Equations (5), (6) and (7) and, consequently, in the calculations based on such equations. In the calculations, velocities of the bubbles were obtained from Figs. 11 to 16 in the same way as for single bubbles. Actually the velocity of a bubble may be influenced by the residual effects due to flow of previous bubbles, such as turbulence and local modifications of bed expansion. These effects may promote collisions disregarded in Equations (5), (6) and (7), as happens when a small bubble collides with a large one rising in front of it. It is not surprising that the discrepancies between the actual and the predicted numbers of passes are considerable in the case of experiments with iron sand ($L_o/\triangle L = 2.0$), silica sand ($L_o/\triangle L = 3.5$) and with low gas flow rates, because in these conditions the channelling of bubbles is more pronounced. Through channelling, the phenomena connected with the above-mentioned residual effects in the beds are accentuated.

Discrepancies between actual bubble velocities and data of bubble velocities of Figs. 11 to 16 may give enhanced values of C. A contrary effect is determined by another phenomenon which has been disregarded in the formulation of Equations (5), (6) and (7): i.e., the breaking off of large bubbles.

Density and size of particles seem to be important in determining this phenomenon. In experiments with glass beads "B" and iron sand, very small bubbles were photographed along with large bubbles. When compared with the number of large bubbles, the fragments were so many that they affected appreciably the average diameter D_{mzz}. This appears in Fig. 5, which is referred to experiments with iron sand. The two cases in which fragments were considered (solid line) and disregarded (broken line) are compared in the diagrams of average diameter vs. distance from nozzle.

Conclusions

The results of our experiments permit us to consider separately the two phenomena, bubble formation above the nozzle and bubble coalescence, occurring in dispersion of gas in solid-liquid fluidised beds.

It is apparent that all the parameters defining the properties of the solid-liquid fluidised systems, in addition to the gas flow rate and nozzle diameter, affect the bubble formation.

On the contrary, bubble coalescence is shown to be dependent mainly on bed expansion. An empirical equation has been written correlating the increase of the average bubble diameter per unit length of the bed with the reciprocal of the degree of the bed expansion.

Besides, a theoretical investigation has been carried out on bubble coalescence which interprets some of the aspects of the phenomenon. The model proposed may also be useful from the practical standpoint, in order to extrapolate results to work conditions which have not been considered in this research.

As far as extrapolation of results is concerned, care should be taken if the container has a size different from that used and if more than one nozzle injects gas in the fluidised bed. Both size of the container and multi-injection have been disregarded in our experiments. However, the effect of both these variables may be accounted for by choosing proper values for S' (Equation (13)). Results of experiments have been worked on to obtain data of specific contact surface between gas and solid-liquid fluidised systems. These data may be of interest in calculations of mass transfer rates in solid-liquid-gas fluidised systems.

Figs. 11-16. *Plots of bubble rise velocities against bubble diameters for different fluidized solid systems.*

Fig. 17. *Determination of* a *and* b *in* $\frac{D_{mZZ} - D_{mo}}{Z} = a\left(\frac{L_o}{\triangle L}\right)^b$

Fig. 18. *Variation of bubble rise velocity in water, oil and in a solid-liquid system with bubble diameter.*

Fig. 19. *Values of the fraction of passes which result in collisions as a function of average bubble diameter.*

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665.54 (43)

GERMAN REFINERY ACTIVITIES

In the U.K., as indeed in the whole of Western Europe, the refining of petroleum is going through a change. Emphasis has shifted from petrol (gasoline) to fuel oils and cracking is on its way out, at least for the time being. Three modern refineries in Germany show the changes dictated by such market demands and by the need for limiting air pollution

REFINING of crude petroleum serves the needs of the market, existing or anticipated, and therefore new processes are being constantly developed to produce as much as possible of what is in greatest demand. The similarity of the words "petroleum" and "petrol" has caused enough confusion, and it is worth stating that from crude oil (petroleum) one can obtain gases, gasoline (petrol), kerosene (jet fuel, turbine fuel, lamp oil) gas oil (diesel fuel), lubricants and various grades of fuel oil. In Europe the market has been less influenced by rapid progress in the automobile industry than in the U.S.A., so the high anti-knock gasoline is not needed in such quantity as it is in the States; there is also greater demand for diesel fuels in Europe than in America.

In Germany the introduction of fuel oils, which form over 50 per cent of all crudes, met an unsuspected resistance from the coal industry, and this product is now taxed to make it less competitive. Having been "brought up" on coal, Germany is finding the flow of crude-derived fuel oils somewhat unsettling, though they provide no more than 11.6 per cent of the total power (1959). The total consumption of crude in Germany is rapidly rising, and estimates for the next five years are the most staggering of all those relating to Europe. Higher than consumption are figures for refinery capacity and those for supplying the refineries with crude, via the two existing pipelines—one to run from Marseilles to Bavaria by way of Strasbourg and Karlsruhe and one from Genoa to Bavaria through Switzerland.

The number of refineries in Western Germany is rapidly rising, since the construction of giants has, apparently, been abandoned in favour of smaller plants located at suitable places to supply areas of industrial development. The existing network of pipelines converges on the Ruhr, where Esso, Petrofina, Shell and B.P. have built new refineries and where old coal-to-liquid fuel plants, based on lignite, have been converted to crude oil refining. The expansion of riparian refineries, which took place soon after the Second World War, has been completed. The Rhineland development brings the second phase to a close; the third phase will be concluded when the Karlsruhe and Bavarian refineries are operating.

Among the Ruhr and Rhineland refineries are also those belonging to the old German coal concerns, which concentrate on the production of petrol by cracking. Their approach to the problem depends on the plant they possess and which, in the case of the Union Kraftstoff at Wesseling,

TABLE I—Petroleum Industry in Germany

New refineries:

1959/61 capacity in m.t.p.a.	
Esso, Köln	3.4
Purfina, Duisburg	3.0
Shell, Godorf	2.5*
B.P., Dinslaken	4.4
Frisia, Emden	1.5
	14.8*

*Due to the expansion of the Shell refinery which will in the near future reach the capacity of 4.0 m.t.p.a., the total will accordingly be 16.3 m.t.p.a.

1963/5 capacity in m.t.p.a.	
Esso, Karlsruhe	4.0
Shell, Ingolstadt	(?) *
Mineral Ölwerke, Karlsruhe	2.0
E.N.I. *et al.*, Ingolstadt	2-3
Esso *et al.* (?), Bavaria	(?) *
	13.0*

*Total refining capacity located in Bavaria is unlikely to exceed 7.0 m.t.p.a.

Total pre-1959: *ca.* 15 m.t.p.a. Total by end of 1962: *ca.* 30 m.t.p.a., Total by the end of 1965: *ca.* 45 m.t.p.a.

Pipeline capacity:[e]

	1960/1	1963/5	1965 and future	
Nord-West Ölleitung (operating from January 1959)	9.0 m.t.p.a.	14.0 m.t.p.a.(?)	20.0 m.t.p.a.[e]	
Rotterdam-Rijn Pijpleiding (operating from June 1960)	6.5 m.t.p.a.	11.0 m.t.p.a.(?)	20.0 m.t.p.a.[e]	
South-European Pipeline (operating from January 1963)[e]	—	10.0 m.t.p.a.	16.0 m.t.p.a.	(Final planned capacity = 30.0 m.t.p.a.)
Genoa-Bavaria (open 1963?)			6.0 m.t.p.a.	
	15.5 m.t.p.a.	33.0 m.t.p.a.	62.0 m.t.p.a.	

Production and demand:	1957	1958	1959	1960	1961	1962	1963	1965
Production (refining)	12.0[a]	15.1[a]	21.7[a]	24.0	—— 28.0 ——		34.0	40.0
Demand (Total)	14.0[a]	18.0[a]	22.5[a]	—— 26.0 ——				36.0
Fuel oils	4.9[b]	7.5[b]	10.0[b/c]	—— 12.0 ——		14.5		17.5
Petrol	—	—	4.5[b/c]					7.0
Diesel fuel	—	—	4.2[b/c]					5.5
Petrochemicals	—	—	0.2[d]					1.2

Sources: [a]Gelsenkirchen Bergwerk AG. [b]German International. [c]Bavarian Government. [d]R.O.W. [e]and all figures beyond 1959 are estimates.

dates from the early- and mid-1950s. The new Esso refinery at Cologne, built in 1958, uses the "hydroskimming" combination of processes, which dispenses with cracking except for the manufacture of petrochemical raw materials. Hydroskimming was first applied some years ago and is used in new Esso plants at Bordeaux, Milford Haven, Rotterdam and also at Whitegate. The development which marks the Shell plant at Godorf, a town between Bonn and Cologne, is significant, because it combines the present-day need for prevention of air and water pollution with a refinery-flow rearrangement. This may be copied in other Shell refineries and, apparently, is a financial success. The approach of these three refineries to the problem of chemicals is also significant, but perhaps it is the result of their policy rather than a change due to a difference in market conditions, though this is undoubtedly relevant too.

The U.K.W., having been concerned with the liquefaction of lignite throughout the Second World War, was bombed so severely that its present plants and activities have little connection with the war days. It still uses coal to make hydrogen and carbon dioxide by a reaction between lignite-derived synthesis gas with steam. This produces more hydrogen and converts CO and CO_2. Additional supplies of carbon monoxide and hydrogen are obtained from heavy oil by the Partial Oxidation process of Shell, which supplements U.K.W. capacity for hydrogen manufacture; they are similarly treated. Oxygen and nitrogen come from a Linden refrigeration plant, and while the former is used as above, the latter is used in the manufacture of ammonia. This and methanol are the two main chemical products; the manufacture of urea, to be sold as fertiliser, has started, using the Staatmijnen 250,000 tpa plant installed recently at U.K.W.

Carbon dioxide is obtained from the same stream as hydrogen by washing out the gases half-way through a compression cycle. This cycle provides hydrogen for the ammonia process at 325 atm or for the refinery at 550 atm, but CO_2 is removed at 28 atm. But for the inclusion of the Shell P.O. process which uses oil, the U.K.W. could not be called a "petrochemical" plant. In some respects, quite the reverse, as the compressed hydrogen derived from coal is sent to the refinery where it is utilised in the process of high-pressure hydrogenation of heavy refining residues.

Also, as a by-product of this operation, H_2S is obtained which, after an alkaline absorption and then release by acid, is converted to pure sulphur by the Claus process. Here H_2S is partly oxidised to SO_2 and this reacts with more H_2S to make steam and molten sulphur.

The refining processes themselves are typical of those which intend to concentrate on maximal production of gasoline and fuels for transport in general at the expense of fuel oil which, in fact, is not produced. Kuwait crude (bought from Shell and Caltex) and indigenous German crude are used. A combination of "main line" and "branch line" processes is used. In their passage along the main line they all begin with distillation of crude which is split into gasoline, diesel fuel and bottoms; bottoms (boiling point over 350°C) are thermally cracked at 450-480°C and 600 psi in a Hydrocarbon-built 550,000 ton/annum cracker and hydrogenated by the Bergius high-pressure process. The hydrogenation process operates in two stages: the first working at 470°C and 8000 psi, the second at 430°C and 4500 psi. The former may be called the "slurry" stage, while the second is done in gaseous phase.

The products of this hydrogenation are again distilled. Tops are thermally reformed in a reformer which was built by the same American firm, Hydrocarbon, in 1953, and has a capacity of 220,000 tons per annum. After that comes polymerisation in a plant built by California Research. Middle fraction is added to other diesel fraction and bottoms are hydrodesulphurised and passed through a Platformer

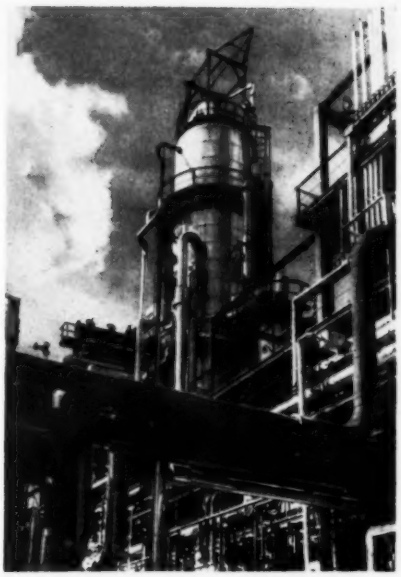

Fig. 1. The main distillation column in this combined refinery/petrochemical plant splits 2 million tons per year of crude into three basic fractions: the middle one of these is Diesel fuel, the tops consist of gasoline and naphtha, while the bottoms are cracking stock.

Fig. 2. The volatile fraction, heavily contaminated with H_2S from the hydrodesulphuriser, is passed through a monoethanolamine wash tower where it is absorbed by this alkali. Upon change of pH the M.E.A. is recovered and recirculated, while H_2S passes to the Claus catalytic unit.

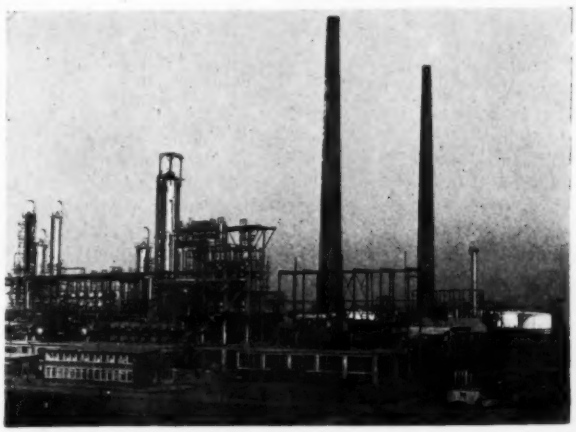

Fig. 3. Platforming is an endothermic catalytic reaction carried out under high pressure of hydrogen. In its course, aliphatic compounds are cyclised and under very severe reforming conditions cyclic aliphatic hydrocarbons may become aromatic. Both reactions liberate hydrogen which has to be bled. Between successive catalytic reaction chambers the hydrocarbons have to be reheated; reforming products must be separated by distillation.

built by U.O.P. This has a capacity of 265,000 t/a and was built by another U.S. firm, Edeleanu, in 1956. The polymerisation unit uses propylene and butylene from the cracker, while ethane and ethylene are supplied to the petrochemical works of Rheinische Olefinwerke by a short pipeline. This works' requirements of ethylene for production of Lupolen (a German version of polyethylene) have recently risen and now include light naphtha from Shell-Godorf which ROW crack themselves.

The Esso refinery produces cracked gases which are piped to three chemical plants in the area. The total length of pipeline is some 55 miles. Among the recipients are Wecker, Ruhrchemie and Holten. The Esso-Köln Raffinerie is supplied by the Nord-West Ölleitung running from Wilhelmshaven to Cologne and Wesseling, also supplying on its way B.P. and Petrofina refineries. At Esso the crude is split into five streams, namely: gases plus light gasoline, naphtha, kerosene, diesel fuel and heavy fuel oil. The first is separated into gases and liquid (boiling points over 35°C, i.e., isopentane plus); the liquid is used for cracking. Gasoline is obtained from reformed naphtha. Virgin naphtha passes through an HDS (hydrodesulphuriser) on its way to a Powerformer, where in the presence of high-pressure hydrogen its octane number is raised.

This process produces more hydrogen, since it is accompanied to a large extent by the cyclisation and formation of aromatics (of high ON) and the hydrogen is used in the HDS plant. This kind of coupling is usual.

The kerosene fraction providing jet fuel is small nowadays, since paraffin lamps and vaporising oil tractors are not common; some of it is used for blending or "cutting back". Diesel/gas oil is also hydrodesulphurised but in another hydrofiner. The conditions of operation are different due to a difference in stock and maximum sulphur permitted in this product, but the HDS process is very versatile (see below). The untreated fraction is partly used for blending with heavy fuel oil to be sold as medium fuel oil, which is heavier than light fuel oil, the next fraction, and the first one to boil below 350°C. The heaviest fraction from the distillation tower is being split at Esso-Köln into HFO and asphalt and this is blown, i.e., slightly oxidised, and so a more viscous, practically solid asphalt is produced. The process is akin to the vulcanisation of rubber or the polymerisation/drying of paints.

A steam cracker in line with naphtha makes petrochemical raw materials. The refinery was designed by Esso Research & Engineering Co. of New Jersey and constructed by German firms, including Lurgi and Guttehoffnunghütte, as sub-contractors to Braun Intern. Corp. It cost DM 235 million.

The problem of keeping the atmospheric pollution to a minimum, especially in an area as highly industrialised as the middle Rhine, has been much in the forefront of the oil companies' thinking. Similar social problems often form some of the difficulties of large concerns where good relationships with local authorities are important. Among these are supplies of water, for which UKW has drilled over 90 wells. Esso uses little water, since air cooling is employed at EKR, and Shell-Godorf uses a cooling tower through which water runs at the rate of 5000 m^3/hr. Make-up for evaporation losses and process water are all the water this refinery needs.

The Shell refinery at Godorf is supplied from the Rotterdam-Rhineland pipeline. This starts near another Shell refinery at Rotterdam, divides at the Dutch-German border into a north and south branch, and the southern branch runs through the Godorf refinery feeding into four bonded tanks along its fence. The pipeline, as mentioned above, ends at Wesseling. The pipeline system in this triangle (Esso, Shell, Wesseling is augmented by the pipelines joining UKW with ROW and Shell-Godorf with ROW, carrying CH_4, C_2H_4 and C_2H_6 gases, liquefied petroleum gases, light tops and naphtha, as well as fuel.

ROW is half owned by Shell and half by BA-und S-F. The Shell refinery will reach its intermediate capacity in 1961. This will be 2½ million tons; it will reach 4 million tons in 1962.

The distillation unit takes 79,000 barrels of Kuwait crude per day. Here it is split as follows: fraction with a final boiling point below 350°C goes overhead to the hydrodesulphuriser and the fraction boiling below that figure and including 2.4 per cent S is not treated at all, being blended into fuels. The hydrodesulphuriser (HDS) has a capacity of 40,000 barrels/day (1.8 mtpa). It is followed by the second distillation tower and the main fractionating column, from which three fractions emerge: bulk overheads, kerosene fraction and bottoms (see below). The overheads are split further into C_4-gas, C_4-liquid and again bottoms, which immediately after are split into light tops and naphtha. Naphtha is sent to a platformer (reformer using platinum catalyst) to improve its ON. The kerosene fraction is not treated further, but the "bottoms" of the main fractional column are later divided into light and heavy gas oil. Blending takes care of the many varied demands for fuels of various viscosities. Products and semi-finished grades are stored in 55 tanks in the refinery and after blending they are despatched. The C_4-streams pass through a monoethylamine solution to remove last traces of sulphur and then they are divided into the various gases. Butanes are sold as caravan, etc., fuels, propane as industrial fuel for special processes and lighter products go to ROW. Shell-Godorf also has a Claus unit. The removal of sulphur from all the fractions boiling above 350°C is the crucial difference between this and all other refineries. This novel step cuts air pollution, removes the need for HDS treatment at various stages, where this generally found in other refineries, removes the need for sulphuric acid treatment of kerosene, the "sweetening" of gasoline by lead plumbate or cupric chloride (a process by which sulphides are converted to polysulphides in the presence of free S). It also is hoped that it will reduce corrosion inside pipes, etc., and, above all, it is claimed to be economical. It may be copied in other Shell refineries. The catalyst for the hydrodesulphurisation is cobalt-molybdenum. Many other features designed to reduce pollution include extra high stacks, the addition of surplus gases to refinery fuel and low-smoking stack. As an illustration of the anticipated needs of the German market, it may be worth noting that the products of the Shell-Godorf refinery, as indeed of EKR, include only 13 per cent of petrol. Other products from Shell-Godorf are 13 per cent of diesel fuel, 2 per cent of turbine fuel and 55 per cent of fuel oil. 7 per cent goes to own consumption and that of other Shell works, 7 per cent is used for petrochemical purposes and 3 per cent is sold as liquid gases. This refinery cost 265 million DM.

It is apparent that in Germany fuel oil demand has risen from *ca.* 30 per cent of the total crude refined to 45 per cent and is expected to reach 48 per cent. Petrol and diesel fuel amount to 19 and 14 per cent respectively but the figures are falling. For the whole of Germany the petrol consumption may still represent (in 1965) 19 per cent, but only 17½ per cent of production. Six million tons of products were imported in 1958, but by 1965 Germany, from an overall importer, will become an exporter of oil products.

Of the processes with great influence on the commercial acceptability of high sulphur crudes, such as those found in the Middle East, hydrodesulphurisation (HDS) is the essential one. This process must precede all the variants of reforming, be it "Powerforming", "Platforming", "Hydroforming" or any of the other trade names, under which individual firms keep their own improvements protected. In the HDS processes the catalyst is Co/Mo mixed oxide supported on alumina, and the process can cope with high

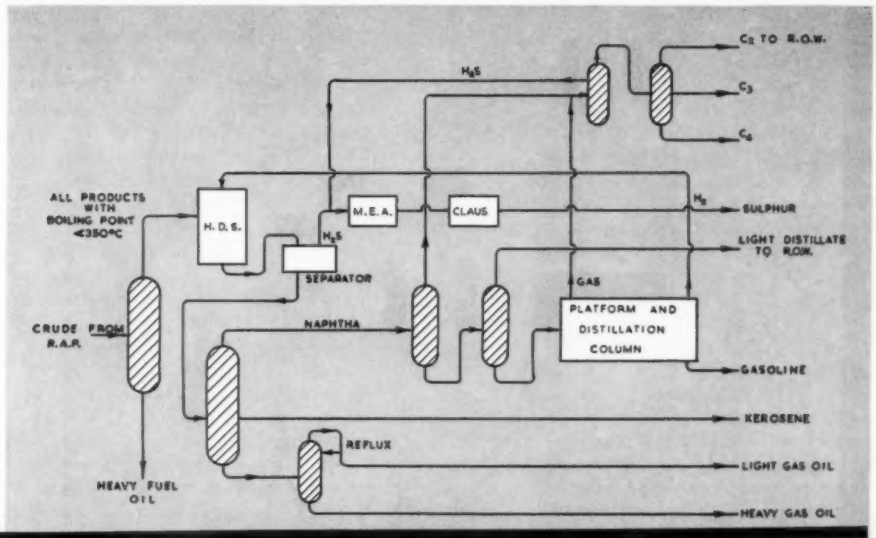

Fig. 4. The Shell refinery at Godorf separates crude into two fractions. The whole of the tops (B.P. under 350° C) is passed through the hydrodesulphurising unit before further treatment. Platformer and distilled unit yield gasoline.

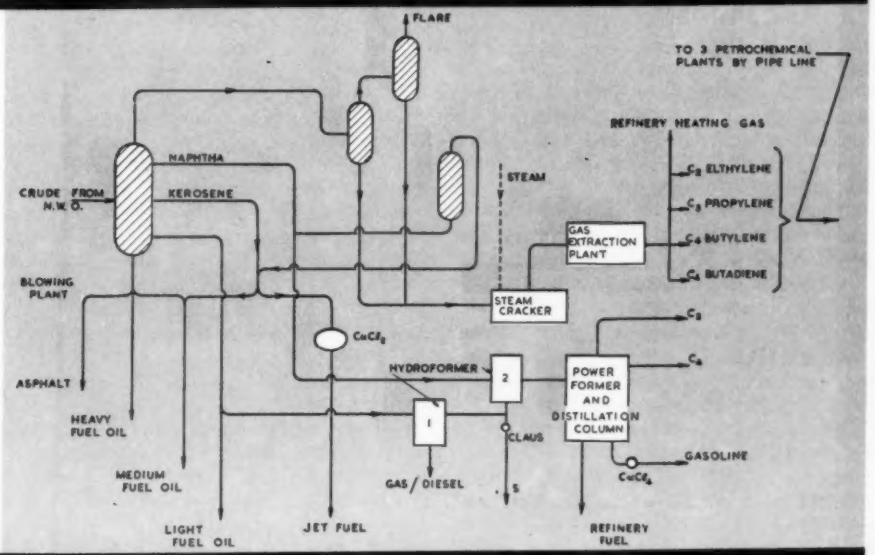

Fig. 5. The Esso refinery at Cologne fractionates crude into five streams which eventually yield ten products. Two hydrofiners are used and reforming, as well as steam cracking, is employed.

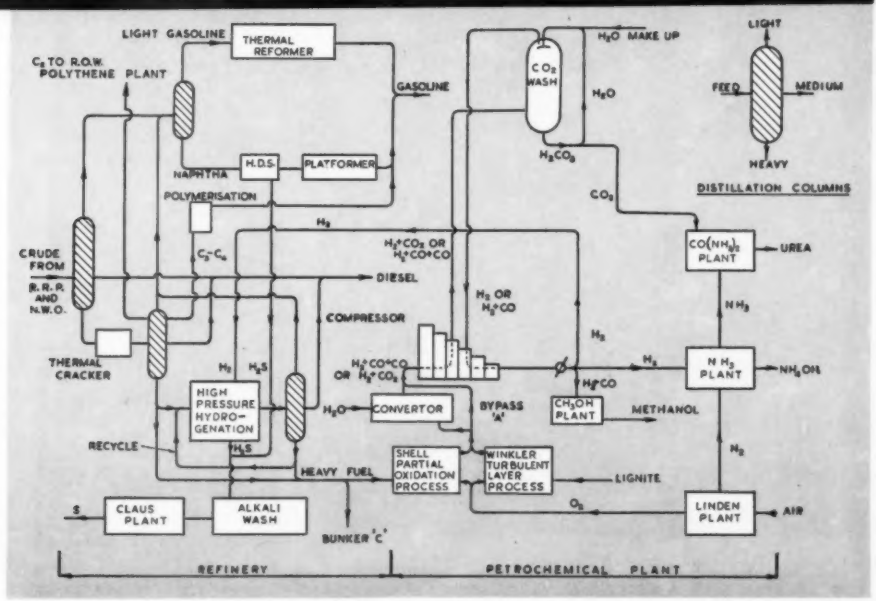

Fig. 6. The UKW refinery at Wesseling is an example of the use of cracking on a very extensive scale. No heavy products are made here. By the use of Shell Partial Oxidation process, some of the heavy fuel is converted to hydrogen, CO_2 and CO, which are used as petrochemical raw materials.

sulphur concentration. At the same time some high ON gasoline is also produced. The charge is mixed with H_2-containing gas recycle and make-up, and after heating it runs down a catalyst bed. Two separators allow H_2 recycle to be free from H_2S. Hydrofining and its variations (Unifining, Autofining, etc.) also perform two functions, i.e., removal of S and upgrading of high-gum (polymerisable) gasolines. Hydrofining itself can be used on a widest variety of stocks. Feed is heated to 550-800°F, mixed with fresh H_2-containing gas and passed down a fixed bed of regenerable catalyst at pressures, depending on severity of treatment desired, from 50-800 psi. Again, two-stage separator is employed. Hydrogen has to be replenished from any process in which it is formed in excess. In Autofining H_2 is generated *in situ* by aromatisation of *cyclo*paraffins. Co/Mo oxides on alumina are again employed. A "knock-out" tower separates fuel oil from the rest of the products, which are then freed from H_2S in an "accumulator". Excessive production of heat is moderated by steam raising through a heat exchanger. Unifining process claims to saturate any olefins present at the same time.

The reforming processes deal with sulphur-free stock and use a platinum-based catalyst, such as Sinclair-Baker which is widely used. The fixed-bed process of Esso takes low ON heavy naphtha and pre-heated H_2-rich gas which enter an endothermic reaction. Three or four reactors are in series with a heater after each one. Pt catalyst in this process requires regeneration every six months. The conditions of the process are that hydrogen-rich gas should be present under pressure, but since the process converts *cyclo*paraffins into aromatics, more H_2 is made. This is generally used in the HDS or hydrofining operation which precedes reforming. Reforming process of UOP claims that any degree of improvement of ON can be achieved with any stock. This is a development of their own Platforming process which does not require regeneration, since the catalyst does not get carbon-coated. Large quantities of aromatics are made and their separation may be a paying proposition for the purpose of petrochemical production. It is reported that the Gelsenberg Benzin AG proposes to make benzene from petroleum using the UOP Udex process, another variant of the reforming process. Powerforming is the Esso process in which catalyst needs regeneration every 80-120 hr, and a spare reactor is needed. Thus one is taken out of operation in turn every four days.

Catalytic and thermal cracking processes yield highly unsaturated products of quite a wide range of molecular weights, so that distillation must follow. Steam cracking, in which partial pressure and residence time of the feed are kept down, provides gaseous stock for the manufacture of polyolefins, also heavy polymerised fuel and gasoline. In any case the feed is mixed with fluidised catalyst and they ascend the reactor together. The feed for cat-cracking is dissolved in heavy gas oil. After cracking, the solids (catalyst) are separated in a cyclone and reactivated by burning off the carbon. The heat of this reaction is utilised for the main cracking reaction itself. The reaction products are separated by fractionation. A modification of this design involves placing of the catalyst regenerator under, instead of by the side of, the reactor. Use of catalyst in spite of all its encumberments is preferred, since it prevents excessive formation of heavy products which occurs at the very high pressures used in thermal process. The other catalyst processes are those based on the use of acids for a Friedel-Craft type reaction. Short olefins are attached to longer chains over sulphuric acid in the catalytic alkylation process and short olefins are polymerised over phosphoric acid in the polymerisation process. Sulphuric acid 98 per cent and *i*-butane are used in the former; propylene and butylene are used for the latter when silica coated with liquid phosphoric acid is used. Many petrochemical processes use this method. Copper chloride and "Doctor" sweetening processes are both of very long standing in the oil industry. Their value is still high, and they are frequently used.

Oil Pollution "Killed" in Open Water

FOR the first time a full-scale operation has been conducted to "kill" oil patches in open water. An operation now concluded at Milford Haven has removed oil pollution from the 100 square mile sea area and the surrounding foreshore and beaches. This was the aftermath of a disastrous fire on the *Esso Portsmouth*, the first ship to discharge at the Esso Co.'s Milford Haven terminal.

Final stage of cleaning jetty is shown by hosing off the Polyclens with water.

A cleaning-up operation conducted by the Esso Co. used 67 men working with launches and sprayers, helicopters and speedboats to spot the patches of pollution. The material used was Polyclens, known to home decorators as a paintbrush cleaner, which emulsifies 200 times its own volume of oil and removes all trace of it. When sprayed on to land pollution the emulsified oil is hosed off with water.

"Killing" the oil on the water is the best means of safeguarding beaches, a problem now being investigated by the D.S.I.R., following an approach to the Government by a number of seaside authorities.

At the time of the fire aboard the *Esso Portsmouth* a spill boom surrounded the tanker at its berth and this contained the oil escaping from the ship's hull. However, the oil within the boom presented a serious fire risk and it was necessary for it to be released with inevitable widespread pollution. Operations have not only cleaned up the affected area, but shore pollution caused by war-time wrecks has also been removed.

Polyclens is a combination of solvents and surface-active agents. It first reduces viscosity, then transforms the oil into a self-emulsifiable mixture easily dispersed in water.

Polyclens is manufactured by Polycell Products Ltd.

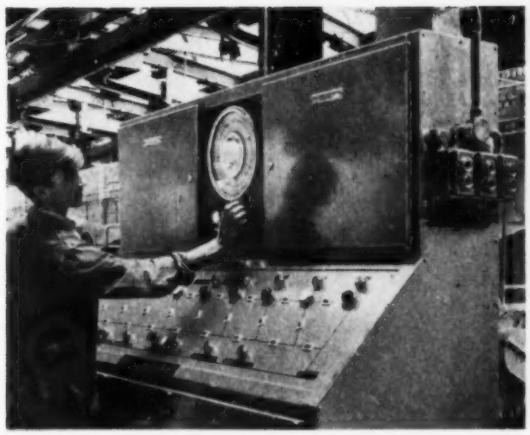

Fig. 1. One of two master control panels controlling automatically the weight of suspension fed to the mono-rail buckets.

DEVELOPMENT OF CHEMICALLY BONDED INDUSTRIAL INSULATION

A SIGNIFICANT development in the field of industrial insulation was recently announced to the Press at the Darlington works of The Chemical & Insulating Co. Ltd. For many years they have been producing 85 per cent magnesia—a combination of 85 per cent light basic magnesium carbonate with 15 per cent long-fibred asbestos—and supplying it as the standard heat-insulation material for all normal industrial purposes.

Now a new manufacturing process has been developed at Darlington to produce 85 per cent super-magnesia, which has interesting features. Instead of the magnesia being merely held together by the reinforcing effect of the asbestos fibre, it is chemically bonded together in such a way that the crystals "grow round" the fibres to give a product of high mechanical strength.

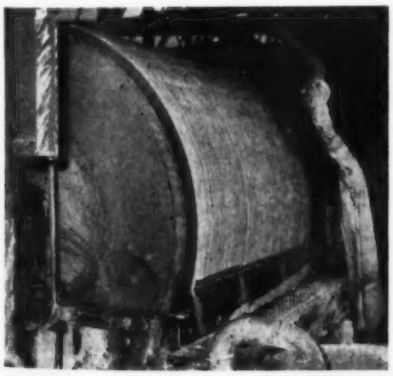

Fig. 2. Rotary vacuum filter discharging 85 per cent super-magnesia as filter cake prior to further processing.

Fig. 3. Preheating of 85 per cent super-magnesia slurry before delivery to moulds.

The process involves the hydraulic setting of magnesium carbonate crystals in a mixture with asbestos fibres during heating under controlled conditions.

The crystals are prepared from magnesium bicarbonate solution. An alternative method is to carbonate magnesium hydroxide suspensions using carbon dioxide gas under controlled conditions of agitation, temperature and concentration. The resultant suspension, containing magnesium carbonate trihydrate crystals, is filtered on a rotary vacuum filter to remove water. The wet cake is removed continuously to a mixing tank, containing water and asbestos fibres, where the entire mass is agitated at a controlled speed to produce a uniform mixture.

After mixing, the suspension is pumped via an automatic weighing device to a container travelling on a mono-rail system. As each empty container reaches the filling station, an interconnecting air-operated solenoid valve permits the suspension to flow into the container until the predetermined weight limit is reached. The container is then despatched along the mono-rail system to the appropriate preheater unit, which is waiting to be filled. The preheater remains in the vertical position when empty, and the travelling container discharges the suspension into it via a hopper chute.

Each preheater supplies one moulding station. A steam injection system fits on to the revolving mouth of the preheater, and steam is passed through the suspension for a time sufficient to raise its temperature to approximately 120-130°F.

When the suspension is sufficiently preheated the steam is automatically cut off. The preheater then tips the heated suspension via the chute into the mould hopper.

The mould cavities are heated by circulating water at high temperature, and the suspension is rapidly converted into a solid mass. As the last stage in the moulding cycle the pieces are pushed out by the pistons and lifted on the bogies for drying. Finally, after drying, the pieces are removed for trimming and packing.

Super-magnesia is rotproof, odourless, fireproof and incapable of corroding metals or harbouring pests. It is most suitable for temperatures up to 650°F. For insulation at temperatures between 650° and 1400°F, Darlington "Paratemp", an insulating material based on calcium silicate, is more suitable.

Photographs supplied by Erwin Wasey, Ruthrauff & Ryan Ltd., on behalf of The Chemical & Insulating Co. Ltd., Darlington.

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FLOW VISUALISATION APPLIED TO A VALVE

The authors review briefly the flow visualisation techniques currently available and then describe an apparatus suitable for the observation of the flow patterns present in models of chemical plant components.

Tests were carried out upon a 2-in. "Y" pattern model valve and the appearance at different cross-sections of the valve body recorded photographically. As a result, certain straightforward modifications to the internal shape of the valve are suggested which improve the flow pattern and reduce the pressure drop by approximately 40 per cent.

Improvements of this kind could also be achieved in some commercially-made valves without complicated modifications

by D. R. OLIVER and Y. MEHRABIAN

THE design of many fluid-flow devices is such that the flow pattern cannot be deduced from a purely theoretical treatment. Even in cases where a theoretical method is available, so many assumptions may be involved that the final predictions are only very approximate. These difficulties have led to the evolution of a number of methods for making flow patterns visible, with the object of either interpreting the flow behaviour of a particular piece of equipment or making modifications to improve the performance. The usual technique is to make a transparent model of the item under investigation, through which either the original or a substitute fluid is passed, the only requirement being that the flow conditions should be dynamically similar. The flow pattern is made visible by the illumination of small quantities of solid, liquid or gas which have been added to the main fluid in order to "trace" out the fluid motion.

The solid tracers which have been used in liquids include aluminium powder,[1, 2, 3, 4, 5, 6] white waxes,[6] polyisobutylene, polyethylene[7] and polystyrene,[8, 9] the latter material being capable of being coated with a fluorescent paint for certain types of work.[7] Tracers of smoke or visible vapours[4, 7] are commonly used in gases; these include kerosene vapour, smoke from burning straw, wood and cigarettes,[10] and ammonium chloride and titanium tetrachloride.[4] For high gas velocities, balsa dust, tufts of wool, chalk dust and glowing coals[7] have been used.

For liquids in laminar flow, liquid streamers of potassium permanganate,[10, 11] coloured inks,[4] aniline dyes,[10] milk and "Dettol"[12] have proved satisfactory, whilst fine globules of oil[13] and other organic substances[4, 10, 12, 14] have been used in both laminar and turbulent flow.

Of gaseous tracers, the most convenient is air.[10, 15, 16, 17] The turbulence naturally present in a fast-flowing liquid breaks the air down into bubbles, and although bubble-size variation and density effects can be troublesome, the method is clean and easy to use.

Two other flow visualisation techniques useful for both liquids and gases are worthy of note. Miniature weathercocks, freely swinging vanes and tufts of silk or wool tied to the model can be practical indicators of flow direction,[7, 10, 35] whilst certain optical effects in fluids can also be employed. In the case of liquids the method applies essentially to laminar flow[18, 19, 20, 21, 22] and is based on the stream-

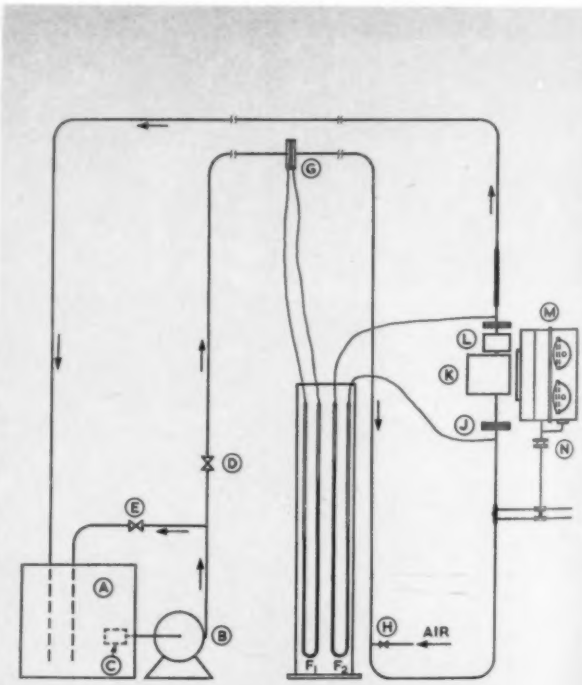

Fig. 1.

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ing bi-refringence induced if small quantities of bentonite,[23, 24] vanadium pentoxide[25, 26] or benzopurpurin[25] are present in the liquid. For gases, the shadowgraph, Schlieren and interferometric techniques[7, 10, 27] may be used, all of which are based on the small density variations which take place in a flowing gas.

The fields of application of flow visualisation techniques are as diverse as the techniques themselves. Thus, the basic nature of flow around solid objects such as flat plates and ellipsoids has been studied,[28] whilst the complex flow patterns obtaining in open-hearth furnaces have also received close attention.[11, 12] Similarly, the flow patterns around finned tubes and banks of tubes have provided useful information,[29, 30] whilst the application of flow visualisation techniques to rocket motor combustion chambers[31] and to the flame tubes of gas turbines[16] have resulted in notable advances in each of these important fields. The problem of flow around aircraft and missiles has frequently been examined by these methods, the technique extending to the measurement of flow patterns around helicopter rotors.[32] In the field of valves, it has been shown that the energy-consuming flow patterns obtaining in disc- and mitre-type valves are such that the pattern changes considerably with valve opening, and in one case 80 per cent of the pressure drop was eliminated by careful design of the "diffuser" section, downstream of the valve itself.

This paper describes how flow patterns were studied in a perspex model of a 2-in. "Y" pattern valve. It proved possible to improve the valve shape and reduce the pressure loss.

Apparatus

A line diagram of the apparatus is shown in Fig. 1. A storage tank (A) feeds water to a centrifugal pump (B) through a form of filter (C), consisting of a perforated copper tube covered with a fine-mesh nylon cloth. The pump having a fixed speed, flow rate is controlled by gate valves (D and E) on the 2-in. I/D discharge line and by-pass line respectively. The mercury manometer (F_1), connected to an orifice gauge (G), is calibrated in terms of the water flow rate by a method involving the direct weighing of the discharge.[33] Air is introduced as flow tracer via a non-return valve (H), and after being broken down into small bubbles is passed through a distribution plate (J) (copper disc with $37\frac{1}{64}$-in.-dia. holes) to ensure uniform distribution. The transparent model, surrounded by rectangular water-filled boxes (K and L) in order to reduce optical distortion, is a 2-in. I/D inclined "Y" valve made from $\frac{1}{4}$-in.-thick perspex, with a screw mechanism of brass. The valve is shown in Fig. 2. The mercury manometer (F_2) was used in order to obtain the pressure drop across the model.

Illumination is provided by two long-range Lucas car lamps,* bolted inside a metal box (M) in such a way that a plane beam of light passes out through two vertical rectangular slots, of width $\frac{1}{8}$ in. and 3 in. apart, located in the side of the box nearer to the model. The slot nearer the lamps is 12 in. long and that nearer the model 10 in. long. In order to move the plane of light horizontally and thus study different vertical cross-sections of the model, the box (M) is mounted on a "horizontal slider" (N), which in turn is supported by an adjustable stand which can be rotated around the axis of the lower 2-in. feed pipe. When photographs are required, the rear part of box M is replaced by a similar structure containing a flash gun.

The model and illumination system is surrounded by a rectangular "darkroom" consisting of a frame draped with black curtains.

Experimental Work

Flow Visualisation

A visual examination of the illuminated model showed that the best angle of observation was about 110° to the direction of the incident beam, the bubbles then appearing as bright points of light. Different cross-sections of the model were studied by the use of the "horizontal slider", three of which are sketched in Fig. 3.

Photography

The intention in this work was to produce photographs in which the bubbles in the upstream approach to the valve appeared as reasonably short streaks; thus a shutter speed of 1/250 to 1/500 sec was required at the higher flow

* Type SLR 700S, 12 V.

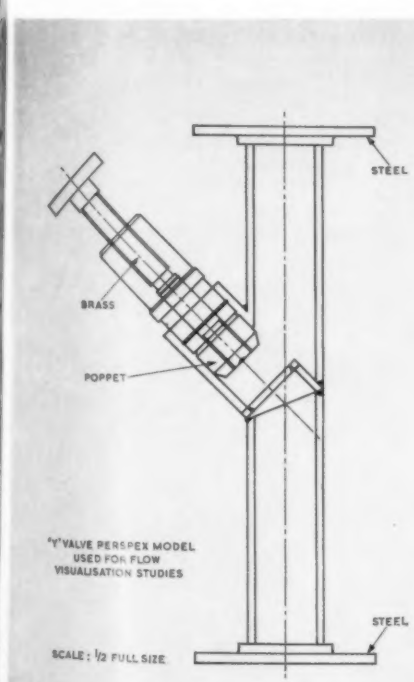


Fig. 2.

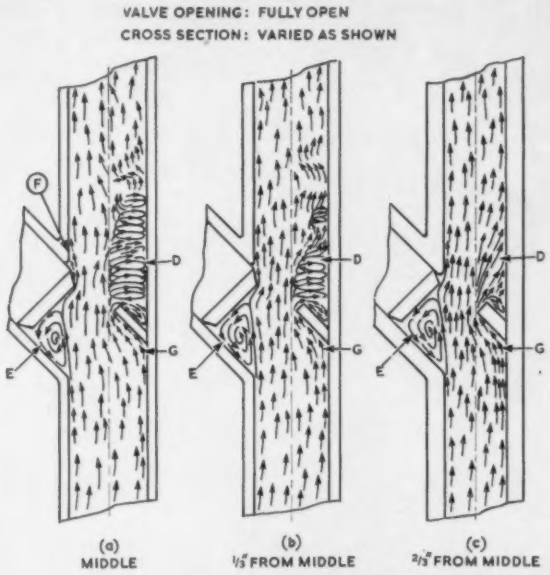


Fig. 3.

Fig. 1. Apparatus used in flow visualisation study of the transparent valve model.

Fig. 2. "Y" valve perspex model used for flow visualisation studies.

Fig. 3. Flow patterns set up in model for different positions of the valve plug.

Fig. 4. Flow pattern in centre cross-section of valve (30 gal/min).

Fig. 5. Flow pattern in cross-section $\frac{1}{4}$ in. from centre (30 gal/min).

Fig. 6. Flow pattern in cross-section $\frac{3}{4}$ in. from centre (30 gal/min).

rates (70 gal/min is equivalent to 8.5 ft/sec). In order to improve the quality of the photographs it was desirable to make sufficient light available to permit the camera to be stopped down to $f = 8.0$ or more.

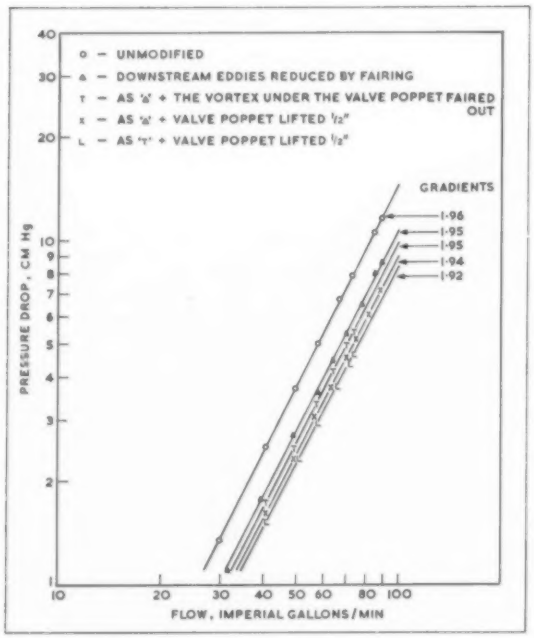
Successful photographs were taken using P.F.5 capless "photoflux" flash bulbs in conjunction with a Rolleiflex $f = 3.5$ camera having an "M" synchronised shutter, i.e., the shutter operates some 17 to 18 milliseconds after the flash bulb has been fired in order to coincide with the period of maximum light intensity. Using this system, an exposure time of 1/500 sec at $f = 8.0$ could be achieved with 35-mm HP3 film, the camera being 3 ft distant from the model.

In order to obtain a complete picture of the flow pattern in the valve, 27 photographs were taken[34] comprising each combination of the following conditions:

Flow rate: 70, 50 and 30 gal/min.

Valve opening: $12\frac{1}{2}$ (fully open), 8 and 4 turns.

Cross-sections: Centre, $\frac{1}{4}$ in. from centre, $\frac{3}{4}$ in. from centre.


o – UNMODIFIED
a – DOWNSTREAM EDDIES REDUCED BY FAIRING
r – AS 'a' + THE VORTEX UNDER THE VALVE POPPET FAIRED OUT
x – AS 'a' + VALVE POPPET LIFTED 1/32"
L – AS 'r' + VALVE POPPET LIFTED 1/32"
GRADIENTS
1·96
1·95
1·95
1·94
1·92
PRESSURE DROP, CM Hg
FLOW, IMPERIAL GALLONS/MIN

The photographs shown in Figs. 4, 5 and 6 correspond to the three cross-sections of the valve, for a flow rate of 30 gal/min and with the valve in its fully open position. In general, the effect of flow rate on the flow pattern was small in the range of flow rates covered. From the photographs the following picture of the flow pattern was built up:

(a) The main eddying region downstream of the valve (region D, Fig. 3) has a convex outer perimeter, so that the eddies extend much farther from the tube wall in the centre than in the other cross-sections. The outer cross-section, in fact, shows definite flow lines with only a small tendency towards eddying.

(b) The vortex formed beneath the valve poppet (region E, Fig. 3) appears to increase slightly in size as the plane of light is moved away from the centre line, suggesting a concave shape for its perimeter.

(c) A slight tendency towards eddying in region F (Fig. 3) was caused by the projection of the valve poppet into the flow path.

(d) The upstream entry to the valve (region G, Fig. 3) did not form vortices.

Modifications to Valve Shape

Modifications, based on the above information, were as follows:

Fig. 8. Flow pattern in valve after modification (a).

Fig. 9. Flow pattern in valve after modification (b).

Fig. 10. Flow pattern in valve after modification (c).

Fig. 11. Flow pattern in valve after modification (d).

Fig. 12. (left). Comparison of the pressure drop of the unmodified 2-in. "Y" valve against those of the modified valves (valve fully open).

Fig. 7. Fairings modifying the valve shape.

(a) Downstream eddies faired out by a piece of shaped perspex (Fig. 7).

(b) As under (a), but also with vortex under poppet faired out by means of a plasticine-filled perspex container (Fig. 7).

(c) As under (a), but with valve poppet withdrawn 0.5 in. from its "open" position.

(d) As under (b), but with valve poppet withdrawn 0.5 in. from its "open" position.

Photographs of the model after each modification are shown in Figs. 8, 9, 10 and 11.

Pressure Drop Measurements

Pressure drop measurements were made on the valve before and after each modification; for this work the distribution plate was removed. The pressure drop of a 2-in. pipe of the same length as the model was also measured and shown to be only 1.5 mm mercury at 80 gal/min. No correction was made for this loss.

Modification	Reduction of Pressure Drop (cm. mercury)	Percentage Reduction of Pressure Drop
(a)	1.35	26.1
(b)	1.70	31.8
(c)	1.95	36.4
(d)	2.15	40.2

The graph of Fig. 1 compares the pressure drops of the various valves at different flow rates plotted on a log-log scale, and the following table summarises the reduction of pressure drop resulting from each modification at a flow rate of 60 gal/min.

Discussion of Results

The most interesting feature of the results is that the elimination of the eddies downstream of the valve itself was a major factor in the final reduction of pressure drop obtained. This illustrates the importance of obtaining smooth flow in the divergent sections of valves.

In contrast, the fairing of the vortex under the valve poppet reduced the pressure loss by only 5.7 per cent (normal fully open position) and 3.8 per cent (poppet withdrawn 0.5 in.). The second figure is probably lower because the fairing was shaped according to the photographs taken before the poppet with withdrawn 0.5 in., a procedure which had the effect of increasing the size of the vortex. It would therefore appear that the figure of 3.8 per cent could be increased a little, given careful fairing design, with consequent improvement of the final pressure drop reduction. The need to design the fairing under the poppet in a manner appropriate to the poppet position shows that this modification can only be used effectively when the valve is being used as an "on and off" control, in which case the fairing must fit through the valve seat when the poppet is in the "closed" position. This may impose some degree of restriction on the effectiveness of the fairing. The shape of the main fairing downstream of the valve should also change somewhat with the valve opening, but the photographs indicate that the effect should be less marked in this case.

The withdrawal of the valve poppet a further 0.5 in. from its "fully open" position reduced the pressure drop by approximately 10 per cent. This is partly due to the elimination of the eddies present in region F (Fig. 3), but mainly due to the increased cross-sectional area of the valve throat. This straightforward modification is one which could be introduced with a minimum of structural alteration, but one which is clearly limited in scope.

Further evidence of the improvement of pressure loss characteristics obtainable by the above methods has been provided by tests of a commercial 2-in. diaphragm-type valve, in which plasticine fairings were used. In the absence of a perspex model, the exact extent of the eddying regions

Fig. 8.

Fig. 9.

Fig. 10.

Fig. 11.

could not be determined, and the eddy shapes were deduced approximately on the basis of the experience of previous tests. It was found that the careful fairing of the convergent entry region of the valve made a negligible difference to the pressure loss; a small downstream fairing reduced the pressure loss by 12 per cent and by fairing an enlargement region within the valve body, and also extending the downstream fairing, an improvement of 40 per cent could be achieved. It is probable that improvements of this order of magnitude could also be obtained with other types of valve.

In the case of the two types of valve tested, the improvement in the flow characteristics could be achieved in practice by straightforward modifications to the castings or to fabricated metal parts. In order to obtain the maximum benefit in valves of limited length, however, it might prove necessary to supply an additional faired extension tube for the downstream end of the valve, which could be used in cases where the pressure losses were required to be kept to an absolute minimum. Even for normal applications, modified valves of the standard length could well give more economical operation than those currently in use.

ACKNOWLEDGMENTS

The authors express their thanks to J. Lucas & Co. Ltd. (Burnley) for practical assistance in the establishment of the experimental method, and to Audley Engineering Co. Ltd., who supplied the model valve. Thanks are also due to Prof. F. H. Garner for his continued encouragement and reading of the proofs.

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Aluminium Pipe in Service

A GOOD idea of the present and potential value of aluminium as a pipework material in the petroleum industry can be gauged from the Esso Petroleum Co.'s experience with an 8-in. aluminium pipeline on their loading jetty at Purfleet, on the Thames Estuary. Recently examined after 12 years' service, during which time it has carried some 430,000 tons of white spirit occasionally contaminated with seawater and with other petroleum products, the pipe was found to have suffered virtually no corrosion while doing a job that with more traditional pipework material would have presented a serious problem.

The aluminium pipe, 8¼ in. O.D., $\frac{5}{16}$ in. wall thickness, in Noral 50SWP alloy (BS 1474: HV9), and the die-cast Noral 125 aluminium alloy (BS 1490: LM16) flanges were supplied by Alcan Industries Ltd. (formerly Northern Aluminium Co. Ltd.). The pipe was installed in 1948, and has since been mainly used to convey white spirit from tankers to the storage tank ashore. Although the pipeline is not normally cleared with seawater, a quantity of seawater has passed through the pipe and a certain amount probably collects in it from time to time. The aluminium pipe has thus been tried in severe conditions.

A Noral 8-in. diameter aluminium pipeline at Purfleet.

Besides demonstrating the pipe's general resistance to corrosion, the 12-year period of service has been a good test of the tightness of the jointing system used. To make the joints, which were developed by Aluminium Construction Ltd., the ends of the pipe were expanded (using a Buck & Hickman tool) into two internal square-cut grooves machined in the cast flanges. The joints were then bolted up with asbestos gaskets. In spite of the penetrating action of the white spirit, and at one place a certain amount of wind-induced vibration, no leakage occurred.

No attempt was made to insulate against bimetallic (galvanic) corrosion at the aluminium-steel joints at either end of the pipe, other than by the use of cadmium-plated bolts and nuts, and some evidence of shallow pitting of the aluminium was found at these positions during an inspection in 1951. Should there be any tendency for corrosion caused in this way to become severe, complete insulation of the aluminium steel joint would, of course, effectively prevent it.

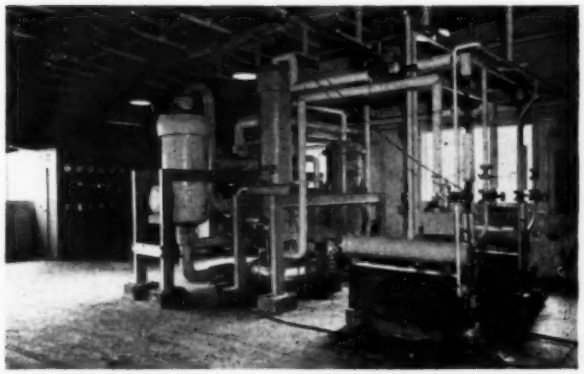
Within the pipe itself, the recent examination showed that a brownish friable deposit had accumulated, but there was virtually no sign of corrosion. Externally the pipe was unnecessarily coated with bituminous paint (in conformity with a general painting scheme) after its first four years of exposure, but no appreciable corrosion had taken place within that time in spite of salt spray and the marine atmosphere. A butt weld had been deliberately included in the line to assess the performance of the Noral 50SWP alloy in the welded condition, and the corrosion resistance of this area was found to be equally good.

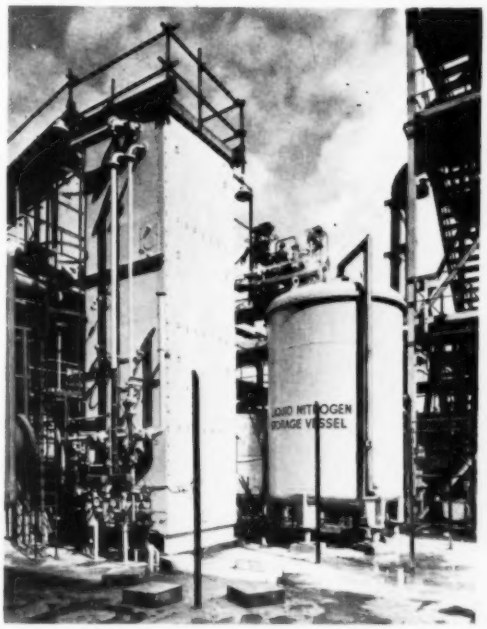
The need to keep service and maintenance costs low becomes increasingly urgent and it is therefore to be expected that the Esso Petroleum Co. will very carefully assess their aluminium pipeline's behaviour. On the strength of the latest examination it will not be surprising if they and others give increasing thought to the potentialities of pipework systems with inherent corrosion resistance.

High-purity Nitrogen Plant

THERE is now a demand for moderate quantities of pure nitrogen for use in blanketing vessels, purging and other purposes. The requirements are large enough to render uneconomical the long-distance transport of compressed gas in bottles or of liquid nitrogen in tanks, and the conventional methods of producing nitrogen on site are not always satisfactory.

Plants based on the combustion of fuels and the subsequent purification of the flue gases show a high cost of utilities, and it is difficult to guarantee a purity of over 99.9 per cent. Normal double-column air separation plants, using high-pressure multi-stage compressors and reciprocating expanders, give rise to occasional maintenance troubles. Moreover, nitrogen is delivered at atmospheric pressure. If nitrogen has to be recompressed, additional compressors have to be installed and the initially pure product may easily be contaminated. Finally, the production of all the nitrogen in liquid form is uneconomical in power consumption.





Photographs of the nitrogen plant supplied by Petrocarbon Developments Ltd.

A New Approach

When the U.K.A.E.A. needed high-purity nitrogen at high pressure, Petrocarbon decided that only an entirely new approach could solve the problem satisfactorily. Experience gained with earlier work on the use of expansion turbines, in connection with oxygen and argon plants, and the distillation of hydrogen for the separation of deuterium, led to the development of a new series of fully automatic plants. The newly developed plant, which is simple to start up and operate, has worked successfully over an extended period in one of the installations of the Production Group of the U.K.A.E.A.

Description of Plant

High-purity nitrogen is produced by the fractionation of air at low temperatures in a column operating at 100 psi, and is delivered to the consumer at this pressure. Compression of the product, with consequent risk of contamination, is thus unnecessary. Nitrogen purity is normally 99.99 per cent, but purer gas can be supplied.

Air is compressed to 110 psi and passes through the purification system and the heat exchangers directly to the column without expansion. Low temperatures are maintained by expanding a part of the compressed air, after its passage through the column, in a turbine of novel design rotating on air bearings at 60,000 rpm.

Capacities of the present series range from 4000 to 40,000 N cu ft/hr of nitrogen and larger units can be provided.

The output of a plant varies automatically with demand, from zero to the upper limit, and power consumption is reduced when the offtake diminishes. If no nitrogen is consumed the plant will "tick over" and will automatically come on stream when consumption is resumed.

For emergency storage and to cover short periods of peak demand, up to 10 per cent of the product can be delivered as liquid, and is then passed to a large vacuum-powder insulated storage tank. If the momentary demand for gas exceeds the productive capacity of the plant, and if, as a result, the pressure in the user's supply line drops, liquid nitrogen is automatically discharged from the tank and vaporised, restoring the pressure to normal and permitting an increased offtake as long as liquid remains in the tank. Should the purity of the product fall below the desired value, production ceases automatically and the plant runs on total reflux until the purity is restored, after which production is automatically resumed.

The consumption of utilities per unit of product nitrogen varies somewhat with the capacity of the plant and with the degree of purity required. For a unit delivering 12,000 cu ft/hr, the power consumption is approximately 120 kW and the cost of the nitrogen, excluding liquid storage facilities, the extent of which depends on the user's requirements, is found to be about 2s. 5d. per thousand cubic feet, inclusive of depreciation and reasonable maintenance charges. This cost figure is an estimate and is not based on operating cost data supplied by U.K.A.E.A.

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THE CONTINUOUS SINTERING OF CALCINED PYRITES

A description of a plant for converting dusty calcines from pyrites roasting processes into a useful product. The heart of this plant is a continuously operating conical sintering machine which uses sulphides as fuel instead of the more usual coke fuel. The layout of a complete plant is illustrated and its advantages over existing methods of treating calcines are enumerated

THE main field for equipment made according to the BOLIDEN-ROLFSEN system is the sintering of the calcines produced from the roasting of various pyrites, particularly by roasting plants of the kind used in the cellulose and sulphuric acid industries. The equipment is low in first cost, occupies little space and can be used therefore even with small roasting units; fuel and labour costs also are very low. Dusty calcines, which are otherwise often valueless, can be converted by this process into a product useful for the iron and steel industries.

Principle of Operation

One fundamental departure of the system, which works continuously in conjunction with a pyrites roasting plant, is that the coke used in conventional methods is replaced by sulphide fuel. The roasted pyrites from the roasting plant is mixed with finely ground pyrites as well as recycled material. The mixture is then fed into the continuously operating grate of the sintering plant. Ignition is by means of an oil-fired ignition chamber, the oil being the sole external source of heat.

The sintering plant is in the form of a truncated cone (Fig. 1), the inclined surface of which acts as a grate and whose top is the exit for the gases. The cone apex angle is 90° and the axis is inclined as 45° to the horizontal.

Thus the upper part of the grate surface (7), to which the material is fed, is horizontal and the diametrically opposite grate surface (4) is vertical. This disposition assists considerably the take-off of the cooled sinter.

The grate is water cooled and is surrounded by annular walls, designed so as to prevent air leakage during the entire process. The water cooling keeps the temperature of the grate tubes down, increases their life and prevents the caking of sinter upon them. The grate tubes are so designed and supported as to avoid destructive thermal stresses even should a tube get blocked and become overheated.

A suction chamber, which is subdivided into several sections, is mounted below the grate. These are connected by pipelines to a manifold (3) in which each section is connected first to a "strong" gas suction line (5) and then to a weak gas line (2). This sequence is carried out automatically as the plant rotates.

The so-called "strong" gas, produced during the sintering process proper, contains up to 5 per cent SO_2. The "weak" gas produced at the conclusion of the sintering process contains little SO_2; it is mainly air, which is used for the final oxidation and cooling of the sinter after the conclusion of the sintering process. The concentration of SO_2 in the "strong" gas can be adjusted, if desired, by changing the ratio of strong gas to weak gas. This is done by means of a sliding valve fitted in the suction manifold.

The suction pressure in each section of the suction chamber is adjusted by easily manipulated dampers (6). As the speed of rotation of the plant can also be regulated within wide limits, the process can be controlled completely either manually or automatically so as to maintain a high SO_2 content suitable for the pyrites roasting plant.

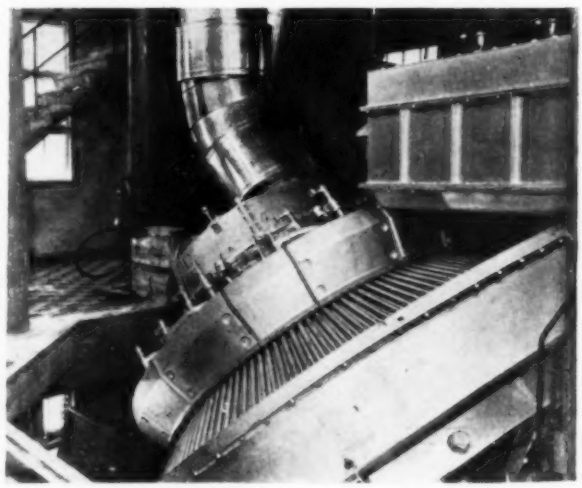

Fig. 1. A view of the upper part of the sintering machine. The ignition chamber is on the right.

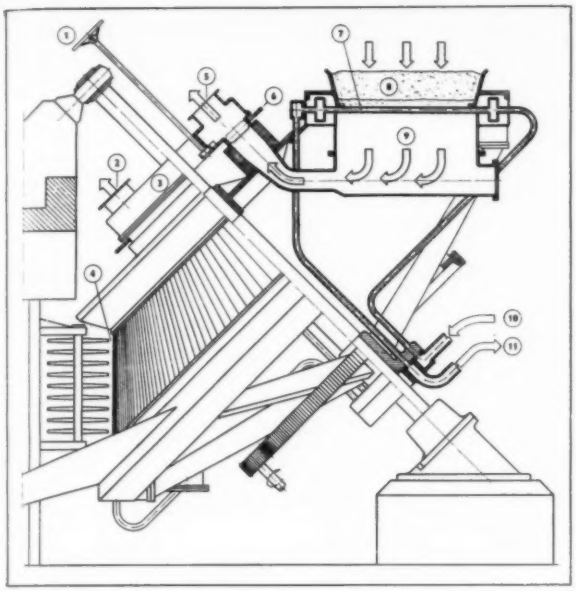

Fig. 2. A section across the machine, showing method of cooling the grate and of preventing air leaks.

(1) *Hand wheel for adjustment of sliding valve which regulates the proportions of strong and weak gases.* (2) *Outlet for weak gas and cooling air.* (3) *Suction manifold.* (4) *Raking and subdivision of sinter cake.* (5) *Outlet for strong gas.* (6) *Damper.* (7) *Water-cooled grate.* (8) *Material for sintering.* (9) *Suction chamber.* (10) *Cooling water inlet.* (11) *Cooling water outlet.*

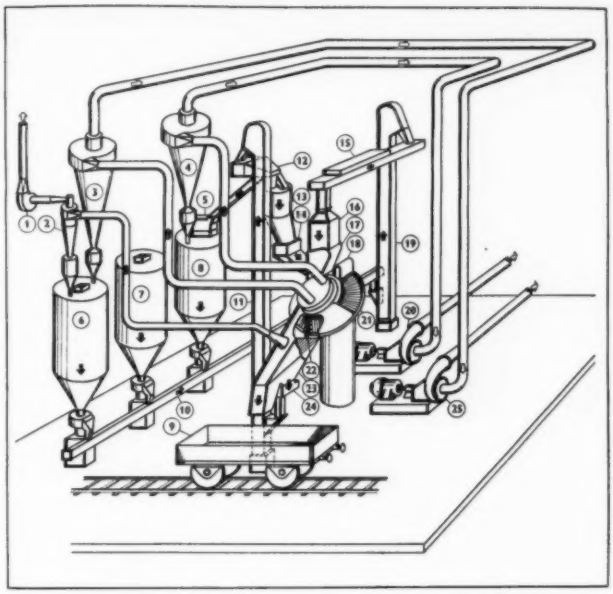

Fig. 3. Layout of a continuous sintering plant.

(1) *Fan for transporting sinter dust to cyclone 2.* (2) *Cyclone for removal of sinter dust.* (3) *Cyclone for cleaning of weak gas and cooling air.* (4) *Cyclone for cleaning sintering gas.* (5) *Crusher for fine crushing of return sinter.* (6) *Storage bin for calcine.* (7) *Storage bin for pyrites.* (8) *Storage bin for return materials.* (9) *Railway wagon for finished first-grade sinter.* (10) *Conveyor for blended material.* (11) *Elevator for return sinter.* (12) *Vibrating screen for return sinter.* (13) *Storage bin for bedding material.* (14) *Feeder for bedding material.* (15) *Mixer for blending and moistening of sinter charge.* (16) *Storage bin for sinter charge.* (17) *Charging equipment for sinter charge.* (18) *Ignition chamber.* (19) *Elevator for blended materials.* (20) *Fan for sintering gas.* (21) *Sintering machine.* (22) *Toothed roller for removal of sinter cake.* (23) *Screen for sinter.* (24) *Crusher for rough crushing of return sinter.* (25) *Fan for weak gas and cooling air.*

Fig. 3 shows the layout of an entire sintering plant. The material fed to the grate consists of bedding material and sinter charge. The purpose of the former, which consists in part of granules and of smaller pieces of sinter, is partly to protect the grate and partly to prevent the fine grain sinter charge from being sucked through the grate. The bedding material is obtained as follows: The finished sinter is screened, and the material which is smaller than the minimum size required for first-class sinter falls through the screen (23), is ground in a coarse crusher (24) and is transported by means of the elevator (11) to a vibrating screen (12). The coarser particles, which are to form the bedding material, are separated here and collected in a storage bin (13), from where it is fed to the grate in a thin layer by means of the feed equipment (14). The finer sinter particles, which pass through the vibration screen (12), pass through the fine grinder (5) and are collected in the bin which contains the return material (8).

The sinter charge itself consists of pyrites calcines and return material as well as some pyrites as fuel. The pyrites calcines, the return material and the pyrites are stored in hoppers (6), (8) and (7) respectively. An adequate supply of this material is delivered to the transporter (10) which feeds the material to the elevator (19). From there it is then transported further to the blender (15). The purpose of the returning material is to provide enough throughway within the sinter charge to enable gases to pass through easily. During the blending operation the material is moistened with water to form an even and loose mix, which is then fed continuously to a smaller storage hopper (16) situated above the feed apparatus (17). During the rotation of the grate, the feed equipment distributes the mix to form an even, loose layer above the previously deposited bedding material. The blend is ignited as it passes beneath the ignition chamber, and the sintering process begins. Ignition is by means of oil.

Air is drawn through the sinter charge by means of a fan (20), and SO_2 formed during the sintering is dedusted by means of a cyclone (4) above the return material storage bin (8) before it passes to the roasting furnace.

Cooling for the sinter cake is supplied by means of a fan (25), which is placed above the pyrites calcines storage hopper (6). Once cooled, the sinter cake is removed and crushed by a rotary rake (22) into suitable sizes for transport and sale.

The advantages of the Celleco sintering equipment are as follows:

(1) It offers a sintered product of very low sulphur content, of the order of a few hundredths of 1 per cent.
(2) It makes sintering an economic proposition for both large and small production units.
(3) The health hazards normally associated with the handling of calcines are eliminated.
(4) Its use involves no serious storage problem.
(5) Freight charges are diminished.
(6) Sulphur losses are virtually eliminated. Any left in the calcines after roasting is the fuel used during the sintering process. The SO_2 produced is utilised.
(7) The operation of the roasting plant is simplified, as it is possible to produce calcine with high sulphur content, without at the same time losing too much sulphur.
(8) Its use provides increased capacity to existing pyrites roasting plants.

622.765.4:661.185

INVESTIGATION OF RESIDENCE TIME OF FLUID IN FROTH FLOTATION CELLS

In connection with a study of fundamental factors influencing the operation and performance of froth flotation cells, the pattern of flow of fluids through laboratory cells of different designs has been studied.

Results show that most types of cell tend to operate as "ideal mixers", the only notable exceptions among those tested being the Forrester cell.

The results of continuous flotation tests in a sub-aeration cell have been correlated with the data on residence time of fluid in the cell, close agreement being obtained between theory and practice.

by A. JOWETT

IT is well known that there are wide variations in the rapidity with which minerals may be recovered in a flotation process. Consequently, one of the most important aspects of the design of a flotation plant is to assess accurately the time of flotation required to give a satisfactory yield of the floating mineral.

If the pulp is fed to a flotation cell of volumetric capacity V litres at a rate of F litres/min, it might seem reasonable to assume that the time, T_N, for which an increment of the pulp is retained in the cell is V/F min. That is, we might assume that the pulp is subjected to flotation for V/F min. In fact, this relationship is only correct in rare circumstances, and probably never applies to an installation of flotation cells.

The error involved in making this assumption can be demonstrated quite simply by reference to the sub-aeration or agitation cell. In a cell of this type the pulp is intimately mixed, and experience shows that it is probably of almost uniform composition throughout the cell; that is, the cell may be behaving as an "ideal mixer". There is evidence available which suggests that the rate of recovery of froth from a flotation cell is governed by a first-order law:

$$C = C_0 \exp(-kT_E) \quad \ldots . (1)$$

or in differential form,

$$-dC/dt = kC \quad \ldots . (2)$$

where C_0 is the concentration of floatable material in the feed, C is the concentration of floatable material in the pulp, T_E is the effective time of flotation or effective residence time in the cell, and k is the rate constant by analogy with chemical kinetics. At equilibrium in a continuous flotation operation, C is constant in any one cell, and the following mass balance equations hold:

Rate of recovery of froth: $-dM_1/dt = kCV$
Rate of feed: $-dM_2/dt = C_0F$
Rate of discharge of tailing: $-dM_3/dt = CR$

where R is the rate of discharge of tailings pulp.

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TABLE I—Flow of Fluid Through a 500-gm Denver Cell
(Pulp volume, V,= 2500 cc; feed rate, F,= 82.0 cc/sec)

Experimental Results		Computations			
Time (secs)	Relative Concentration (Ct)	Ct^{100}	$Ct^{100}\,\Delta t$	$\sum_0^t Ct^{100}\,\Delta t$	FT/V
5	10.0	3.744	18.72	18.72	0.16
10	7.1	2.658	13.29	32.01	0.32
15	5.9	2.210	11.05	43.06	0.49
20	5.0	1.872	9.36	52.42	0.66
25	3.86	1.446	7.23	59.62	0.82
30	3.30	1.236	6.18	65.80	0.98
35	2.66	0.996	4.98	70.78	1.15
40	2.30	0.862	4.31	75.09	1.31
45	1.98	0.740	3.70	78.79	1.48
50	1.84	0.690	3.45	82.24	1.64
55	1.60	0.580	2.90	85.14	1.80
60	1.38	0.518	2.59	87.73	1.97
70	0.96	0.359	3.59	91.32	2.30
80	0.72	0.270	2.70	94.02	2.62
90	0.47	0.176	1.76	95.78	2.95
100	0.29	0.109	1.09	96.87	3.28
110	0.20	0.075	0.75	97.62	3.61
120	0.10	0.057	0.57	98.19	3.94
130	0.09	0.034	0.34	98.53	4.26
140	0.05	0.019	0.19	98.72	4.59

TABLE II—Correlation of Denver Cell Data with Residence Time Theory

	Flotation Test No.	Feed Rate (cc/min)	Pulp Volume (cc)	Tailings Discharge Rate (cc/min)	Actual Yield (%)	Actual Yield of Floatable Material (%)	Theoretical Yield (%)
Series 1 (k= 1.60 min^{-1})	1	5920	2510	5015	35.1	43.8	44.4
	2	4370	2500	3479	42.3	52.8	53.4
	3	3235	2497	2478	51.2	63.9	61.8
	4	3122	2462	2370	52.3	65.6	62.4
	5	3042	2462	2358	50.0	62.3	62.6
	6	2100	2438	1583	57.3	71.5	71.2
	7	1860	2438	1385	58.7	73.3	73.8
Series 2 (k= 3.15 min^{-1})	1	6420	2510	4920	55.8	61.2	61.6
	2	6270	2510	4715	57.5	62.8	62.6
	3	4920	2500	3612	64.5	70.6	68.6
	4	4880	2500	3552	64.4	70.4	68.8
	5	3585	2462	2426	68.6	75.1	76.2
	6	3585	2462	2410	70.2	76.7	76.3
	7	2672	2438	1618	74.8	82.0	82.6
	8	2640	2438	1726	75.1	82.2	81.7

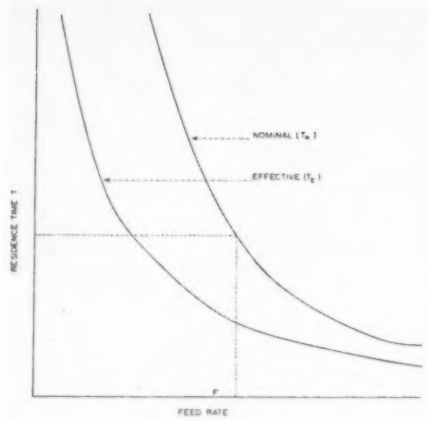

Fig. 1. Relationship between nominal and effective residence times.

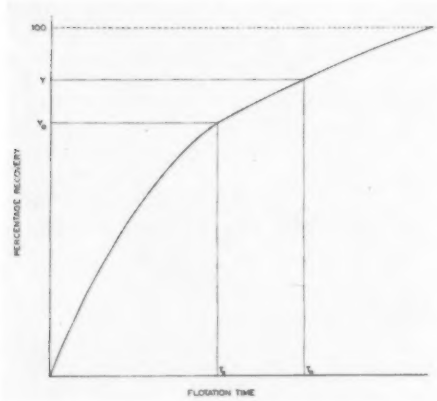

Fig. 2. Yields corresponding to different residence times.

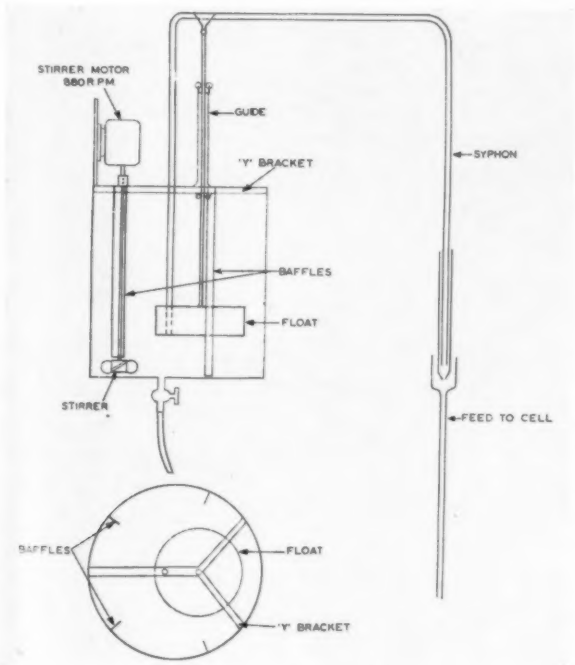

Fig. 3. Continuous feed tank.

Since dM/dt is zero at equilibrium (i.e., mass of solid in the cell is constant):

$$C_0F = CR + kCV \quad \ldots.(3)$$

from which $$C = C_0/(R/F + kT_N) \quad \ldots.(3a)$$

From Equations (1) and (3a),

$$C/C_0 = \exp(-kT_E) = 1/(R/F + kT_N)$$

$$T_E = 1n\,(R/F + kT_N)/k \quad \ldots.(4)$$

(N.B. Where there is only one effluent from the reaction vessel, as in an ordinary chemical process, R and F are equal and the expression simplifies to one involving time factors only.)

This equation gives us a relationship between the nominal residence time, T_N, and the effective residence time, T_E, in an ideal mixer, as illustrated in Fig. 1. At any feed rate, F, the effective residence time is much less than the nominal residence time. Converting this into practical terms, we have the situation illustrated in Fig. 2, where miscalculation of the required residence time causes a significant loss of valuable mineral; this has resulted from an under-estimation of the volumetric capacity required of a bank of flotation cells.

The preceding calculations have been made to illustrate the difference between the nominal and effective residence times. It is not certain that the sub-aeration type cell gives ideal mixing, and many other types of cell certainly do not. Consequently, the relationship between the nominal and effective residence times is hardly likely to be as simple in practice as is indicated by the above calculations made for an ideal mixer, and in a majority of cases the relationship will probably be found to be empirical.

To obtain relationships between nominal and effective residence time, series of tests have been carried out on several different designs of laboratory flotation cell and on a bank of four No. 7 Denver cells. The characteristics of the flow of water through the cells have been investigated, the nominal and effective residence times have been compared, and the results have been analysed to compare the cells with an ideal mixer.

The results of continuous laboratory flotation tests in a Denver laboratory batch flotation cell and a Fagergren laboratory cell, both modified for continuous operation, have been correlated with residence time investigations in the same cells. The correlation indicates the possible value of both residence time tests and flotation rate tests for obtaining capacity data for flotation plants.

Experimental Method

The characteristics of the flow of fluid through a vessel or apparatus can be determined quite simply. The fluid is passed through the apparatus at a steady rate of flow, and when equilibrium conditions obtain a batch of tracer (e.g., dye, acid, salt, foreign gas, etc.) is added to the stream of fluid at the feed point. Samples of the effluent from the apparatus are collected at specified time intervals, timing commencing when the tracer is added. The samples are analysed, and the concentration of tracer in the effluent is plotted against time. The resultant curve is similar to a frequency distribution curve, the concentration of tracer at any instant being a measure of the probability that an increment of the feed will be retained in the apparatus for a certain period of time.

This technique has been used by chemical engineers to determine the characteristics of the flow of fluid through reaction vessels of various types (e.g., aeration vessels and sludge tanks); such information has been found essential to provide adequate design data. A flotation cell is fundamentally a form of reaction vessel, and consequently a detailed knowledge of the flow characteristics and time factors in-

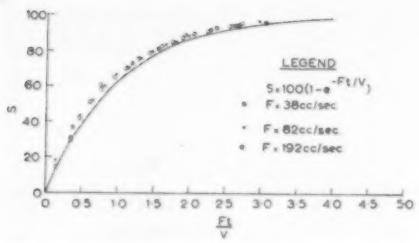

Fig. 4. S diagram for the Denver cell.

TABLE III—Correlation of Fagergren Cell Data with Residence Time Theory (V = 2600 cc; $k$mean = 0.80 min^{-1})

Flotation Test No.	Pulp Density of Feed (gm/l)	Feed Rate (cc/min)	Tailings Discharge Rate (cc/min)	Actual Yield (%)	Theoretical Yield (%)
1	74.2	2032	1517	59.6	57.8
2	90.2	2006	1465	58.7	58.7
3	108.0	2024	1372	61.8	60.3
4	136.0	1955	1232	62.7	62.8
5	143.0	1986	1265	60.9	62.2
6	156.0	2026	1194	63.8	63.6
7	183.0	1940	1016	66.0	67.2

volved in a particular cell may be expected to constitute an important feature of its design.

In the present series of tests, water was fed to the various cells from a constant head device, and when equilibrium conditions had become established about 20 cc of a strong solution of methylene blue were poured quickly into the stream of liquid entering the cell. Samples of effluent were collected at regular time intervals until all the dye had been discharged from the system, a final sample then being taken as a blank. The relative concentration of dye in each sample was measured with a colorimeter. The flow characteristics were established by carrying out tests at different feed rates on single cells and on groups of 2, 3 and 4 cells in series.

Flow Through Cells of Different Designs

This series of residence time tests was carried out as part of an investigation of flotation cell design. Four cells, all laboratory models and each of fundamentally different operation, were investigated. These were:

(i) 500-gm Denver laboratory cell (sub-aeration type);
(ii) 10-litre Forrester laboratory cell (cascade type);
(iii) 4.5-litre single-compartment Macintosh laboratory cell (deep mat-type cell);
(iv) 2-litre shallow mat cell.

Some tests were also carried out on a Fagergren laboratory flotation machine, which is also a sub-aeration cell. All the cells were suitably modified for continuous operation by provision of tailings discharge pipes, pulp level controls and mechanical froth scrapers where required.

The apparatus used for feeding water to the cells was a floating syphon-feed arrangement of the type developed by SCHUHMANN (1942); this apparatus had been contructed and used for flotation rate tests with the same cells. The general arrangement is indicated in Fig. 3. The syphon tube consists of a copper tube, threaded at the dscharge end; the syphon head may be adjusted by screwing on to the discharge an appropriate length of tube.

The results (and subsequent computations) from a typical test are given in Table I. This particular table relates to one of the tests carried out on the 500-gm Denver cell, and is explained fully below.

Analysis of Results

Various methods are available for the analysis of results obtained from residence time tests. One of the most useful is that developed by DANCKWERTS (1953), which compares the actual results with those which would be obtained theoretically from an "ideal mixer". An ideal mixer may be defined as a stirred vessel in which an increment of fluid feed is immediately dispersed uniformly throughout the vessel; it follows that an effluent must have the same composition as the fluid in the vessel.

MACMULLIN and WEBER (1935) have shown that, for an ideal mixer, the fraction, S, of reactants (dye) which has left the system after any time, t, is $(1 - e^{-Ft/V})$. For a number of ideal mixers in series the following general equation applies:

$$S = [1 - e^{-Ft/V}(1 + Ft/V + F^2t^2/2V^2 + \ldots F^{(n-1)}t^{(n-1)}/V^{(n-1)} \cdot (n-1)]$$

where n is the number of reactors (i.e., cells in series).

The reading (E) given by the colorimeter is directly proportional to the concentration of dye (C), and the concentration can be read direct from a simple calibration curve.

It is not easy in these experiments to ensure that the same amount of dye (S) is added in every case. Nor is it necessary to aim at adding always the same amount, since all the results can be brought to a common denominator in terms of both quantity of dye and time factors by simple computations.

The area, S_1, under the curve C versus t is directly proportional to the total quantity of reactant (i.e., dye) fed to the cell:

$$S_1 = \int_0^\infty C \cdot dt = \sum_{t=0}^{t=\infty} C \cdot \triangle t$$

where $\triangle t = (t_{(n+1)} - t_{(n-1)})/2$. Calculation has shown these two relationships to hold for the results to within the estimated limits of experimental error.

The common denominator for quantity of dye is easily obtained by letting S_1 be 100 arbitrary units, so that any value of C_t then becomes:

$$C_t^{(100)} = 100.C_t/S_{1(\text{actual})}.$$

A list of adjusted relative concentrations is given in column 3 of Table I. All these readings can be brought to a common denominator on the time scale also by plotting them against Ft/V instead of t.

The product, $\sum_0^t C_t^{(100)}.\triangle t$, plotted against Ft/v gives the curve (the S curve) which has been used by DANCKWERTS to obtain a measure of the degree of mixing obtained in a system.

Fig. 4 illustrates the theoretical curve:

$$S \text{ (per cent)} = 100(1 - e^{-Ft/V})$$

along with experimental results obtained for the 500-gm Denver cell at a series of feed rates. Fig. 5 shows the same diagram for the other four cells; the Fagergren cell was tested at only one feed rate, since it was so obviously an ideal mixer, and the Forrester cell could only be tested at one reasonably high feed rate on account of its larger capacity than the other cells.

Interpretation of Results

It is important at this stage to consider the significance of the relative positions and shapes of the observed residence time curves and the theoretical curve for an ideal mixer. This has been dealt with in great detail by DANCKWERTS. Briefly, the ideal mixer represents one extreme in reaction vessel design; the other extreme is "piston flow", in which

an increment of feed (reactant) flows like a piston from feed to outlet (say of a tube) with no longitudinal mixing, although lateral mixing may occur. Intermediate are a large number of reaction vessels with some tendency for piston flow and also some degree of longitudinal mixing. Short-circuiting of feed is zero in a piston flow vessel, and generally at a maximum in ideal mixers.

The exception arises in vessels which contain dead space, in which case short-circuiting will be greater than for a similar vessel with no dead-space, since the feed can go almost direct from inlet to outlet.

Each form of fluid flow has its own characteristic residence time curve and, by comparing the actual curve with that for either an ideal mixer or a piston-flow vessel, factors representing degree of mixing, short-circuiting and dead-space can be determined. DANCKWERTS gives details of the methods of comparing vessels with an ideal mixer.

It will be apparent from the results presented that the Fagergren cell and the Denver cell approximate very closely to ideal mixers; also repeat tests at different feed rates show that the flow characteristics of the Denver cell remain very constant.

The results with the shallow mat cell are indefinite and various repeats of the tests suggested that the flow characteristics of this cell were unstable.

The Forrester cell and the Macintosh cell represent the extremes in the cells tested. The Forrester cell is a case of the transition between the ideal mixer and piston-flow vessel, and in this cell short-circuiting of pulp is the least of all the cells tested. On the other hand there is some indication that the Macintosh cell (the deep, mat cell) contains some dead-space, and so short-circuiting of pulp is at a maximum in this cell. This might be anticipated with this design of cell, in which agitation is weak and the ratio of cell depth to width is large; pulp fed in at the surface at one end of the cell could easily flow across the cell to the discharge without being thoroughly distributed through the whole volume of pulp.

These points are of great importance in connection with the yield of floatable material obtained from a flotation circuit. If the degree of short-circuiting is high, a high yield may only be assured by installing several cells in series to assure a sufficient residence time for all increments of pulp, which is the usual practice in the case of the ideal mixers. If the cell tends to approximate more to a piston-flow vessel, fewer cells of larger volumetric capacity can be used; this will probably result in a marked decrease in capital costs.

The Flow of Pulp Through Sub-aeration Cells in Series

Following on the preliminary investigations with single cells of different designs, it was decided to check experimental evidence for ideal mixing in sub-aeration cells along with the MCMULLIN and WEBER theories for ideal mixers in series.

Experiments were carried out on a bank of four Denver No. 7 sub-aeration cells in series. Water was fed to the cells from a simple constant-head apparatus fed from the mains water supply; the flow rate was adjusted simply by screwing nozzles of different diameters on to the pipe discharging into the cells. The cells had been constructed so that the overflow from one cell passed down an open pipe into the impeller zone of the next cell. Water could be fed from the constant-head apparatus direct to the appropriate pipe to give 1, 2, 3 or 4 cells in series; the methylene blue solution also could be poured into the pipe, thus ensuring thorough initial mixing.

The tests were carried out at four different feed rates for each case of 1, 2, 3 or 4 cells in series. It was observed that the pulp level in the cells varied with the feed rate, and so the pulp volume was measured at each feed rate. The results of these tests have been recalculated to the basic standards used by DANCKWERTS and plotted to compare with the theoretical curves given by the MCMULLIN and WEBER relationship for stirred reactors in series. The experimental results fit the theoretical curves closely for all the combinations of cells in series; by way of example, the results for 2 cells in series are given in Fig. 6.

Various workers in the field of flotation kinetics (SCHUHMANN; BROWN and SMITH, 1954; DE BRUYN and MODI, 1956) have implicitly assumed that sub-aeration cells do behave as ideal mixers; the present tests prove the validity of their assumptions so far as the liquid is concerned. In relation to flotation kinetics it is most interesting to correlate these experiments on flow characteristics with determinations of froth flotation rates.

Correlation of Residence Time Theory with Data from Continuous Flotation Tests

Although the experiments have proved that the Denver and Fagergren laboratory cells behave as ideal mixers so far as fluid flow is concerned, it might be argued that the degree of turbulence is nevertheless insufficient to create ideal mixing of the solid particles suspended in the fluid; i.e., there may be some degree of sedimentation in the cells.

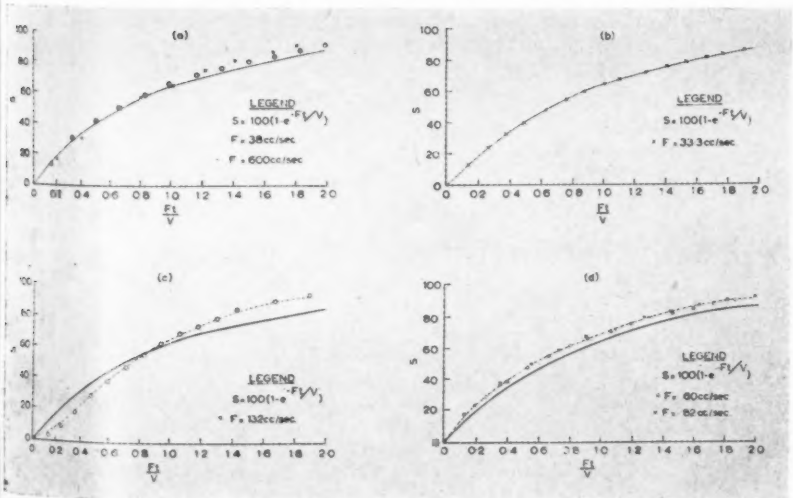

Fig. 5.
(a) S diagram for the Mat cell.
(b) S diagram for the Fagergren cell.
(c) S diagram for the Forrester cell.
(d) S diagram for the Mackintosh cell.

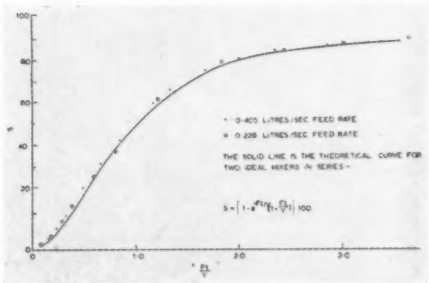
0·405 LITRES/SEC FEED RATE
0·228 LITRES/SEC FEED RATE
THE SOLID LINE IS THE THEORETICAL CURVE FOR TWO IDEAL MIXERS IN SERIES
S
Ft/V

Fig. 6. Flow of water through Denver cells (two cells).

It is possible to test this contention by carrying out continuous flotation tests and correlating the actual yield obtained with the theoretical yield assuming ideal mixing of all material fed to the cell.

In parallel with the residence time tests on the 500-gm Denver laboratory cell and the Fagergren laboratory cell, small-scale continuous flotation tests have been carried out; these results have provided further evidence of first-order kinetics for the flotation reaction (JOWETT and SAFVI, 1960). The mass balance relationship given in Equation (3) only holds for a first-order process taking place under conditions of ideal mixing. The theoretical yield in a continuous flotation operation is given by the expression—

$$Y_t = \frac{\text{Rate of recovery of solid as froth}}{\text{Rate of feed of solid}}$$
$$= kCV/CoF$$
$$= kCV/(CR + kCV) \ldots \text{from Equation (3)}$$
$$= kV/(R + kV)$$

This expression for yield only holds if the flotation operation is proceeding by a first-order process in a cell in which there is ideal mixing. Consequently, if feed rate, tailings discharge rate, pulp volume and a rate constant (being a measure of floatability of the feed) are known, the theoretical yield can be calculated. Table II presents the results of a series of continuous flotation tests using the Denver cell and Table III gives similar results for the Fagergren cell. Feed rate, tailings discharge rate, actual yield and rate constant are all experimental data; the theoretical yield is calculated by substitution of the measured rate constant in the theoretical equation given above. The correlation between the theoretical yield and the actual yield of floatable material is good.

Tests with Denver and Fagergren Flotation Cells

The two series of tests with the Denver cell were carried out at different feed rates with a constant pulp density of the feed. Not all of the feed was floatable, and the maximum percentage recovery obtained in a batch test was taken as representing the percentage of floatable material in the solids fed to the cell; this was 80.1 per cent for Series 1 and 91.5 per cent for Series 2. Consequently, the theoretical yield does not correlate with the yield of total solids fed to the cell.

The tests in the Fagergren cell were conducted with a series of pulps of different solids content, all fed at nominally the same feed rate. The conditions of testing were such that the proportion of non-floatable material present appeared to be zero; this point is discussed fully by JOWETT and SAFVI (1960). Consequently, the theoretical yield correlates with the yield of total solids fed in this series of tests.

It would appear from these correlations that a method for the prediction on a sound theoretical basis of flotation cell capacities and results might be developed for a flotation cell which behaves as an ideal mixer. Much more information concerning R and k will probably be required, however, in relation to other flotation variables such as impeller speed, rate of aeration, etc., before the theory might be applied with any degree of confidence to the calculation of flotation plant capacities.

Also, it must be stressed that this simple relationship can only hold for cells which behave as ideal mixers. The residence time tests have shown that some cells do not, and it is a tedious and much more complex operation to calculate an effective residence time for cells of this type. As well as involving a graphical integration of doubtful accuracy, assumptions must be made concerning the rate constant which may be of doubtful validity in view of the present state of knowledge of flotation kinetics; it is important to realise that a rate constant is a complex function influenced both by inherent properties of the material being floated and by the conditions of flotation in a particular cell. A first-order process has to be accepted so that calculations of flotation rate can be made in terms of the "active mass" of material in the cell. That is:

$$\text{Rate of flotation} = k.M$$

where M is the total mass of floatable material in the cell at equilibrium.

If the cell is an ideal mixer, M is equal to CV, but for any other type of cell (e.g., the Forrester cell) the expression for M becomes much more complex, since the concentration of floatable material is not uniform throughout the cell. Also, for non-ideal mixing, C will represent the probable minimum concentration in the cell (unless dead-space is present); consequently, the theoretical expression for yield would give a figure different from the actual yield.

If there are n zones each of volume, v, within the cell and each with a different concentration, c, of floatable material, the overall rate of recovery ($k.M$) will be:

$$\text{Rate of flotation} = kv. \sum_1^n c$$

The mass balance relationship at equilibrium is then:

$$CoF = RC + kv. \sum_1^n c$$

and the theoretical yield is:

$$Y_t = \frac{kv. \sum_1^n c}{RC + kv. \sum_1^n c}$$

For the ideal mixer:

$$c_1 = c_2 = \ldots c_n = C$$

and so:

$$Y_t = k. \sum_1^n vC/(RC + k. \sum_1^n vC)$$
$$= kVC/(RC + kVC)$$
$$= kV/(R + kV)$$

The work described in this paper has been carried out as part of a broad investigation of the flotation process and in particular of the design of flotation cells. These experiments on flotation kinetics and the flow characteristics of pulp have clarified many obscure points relating to the mechanics of flotation, and provide a valuable reference point for any further work on flotation cell design.

ACKNOWLEDGMENTS

The author gratefully acknowledges the encouragement and advice given by PROFESSOR J. T. WHETTON, D.S.O., O.B.E., M.C., T.D., M.Sc., and also the assistance of colleagues and students in the Department of Mining. Financial assistance from the National Coal Board is also gratefully acknowledged.

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EXPERIENCES IN FLUIDISATION

A Symposium on Fluidisation was held at Adelaide, February 15 to 17, 1961, under the auspices of the South Australian Branch of The Royal Australian Chemical Institute.

One paper presented studies on particle residence time determinations in fluidised reactors, using magnetically tagged particles, and another considered agglomeration: the factors which affect it, the means of controlling it and methods of testing for it. The other papers were concerned with specific processes and reactions, namely, sulphiding and chlorination of ilmenite, roasting of zinc plant residues and copper concentrates, carbonisation of coal. Design and operation data were presented in some cases.

Determination of Particle Residence Time in a Fluidised Bed *by K. McG. Bowling and A. Watts*

MAGNETICALLY tagged char particles were used to determine the distribution of particle residence times in a 2 in. diameter laboratory-scale reactor, fed continuously with fresh solids.

"Jump-signal" and "step-signal" type experiments were conducted. In the former, tagged particles were added over a very short time and the outlet concentration determined over a much longer time. In the latter type, similar determinations of outlet concentration were made, after making a step-wise increase in the concentration of the feed.

Characteristics of the distribution such as hold back, segregation and medium residence time were evaluated and compared with the three model cases of piston flow, perfect mixing and perfect by-passing, in a series of figures. These figures also indicate the fraction of bed particles having less than a given residence time, the residence time for a specified fraction of the bed and the fraction of original bed having a residence time within a given percentage of the calculated mean.

The actual distributions of residence times obtained for the cases studied were similar to those theoretically obtainable with a perfectly mixed bed. The results were practically unaffected by differences in feed rates and bed depth and by the presence, or absence, of a gas distribution grid at the bottom of the bed.

Two special flow conditions were also investigated, namely:

(a) admission of particles at the top of the bed, and

(b) "spouting", where a large proportion of feed material travels quickly, up the axis of the bed, to the fountain-shaped region at the top, a portion of it leaving the bed without further treatment. In both cases the residence time of a proportion of the feed particles was reduced and the distribution of residence times was thereby changed.

The experiments showed that uniform residence times cannot be achieved in normal single-stage fluidised beds.

The Sulphiding of Ilmenite in a Fluidised Bed Reactor *by A. B. Whitehead and C. J. Restarick*

A NEW process for the conversion of ilmenite to granular rutile in two main reaction steps (first suggested in 1956) is being evaluated in Australia. In the first-stage reaction ilmenite is reacted with sulphur vapour at high temperature:

$$2\,FeO\,.\,TiO_2 + 3S \rightarrow 2\,FeS + 2\,TiO_2 + SO_2$$

The iron sulphide is then partially removed from the skeletal lattice of TiO_2 by hydrothermal oxidation with oxygen.

To provide data for preliminary plant design and economic assessment, both stages of the reaction have been studied by the Chemical Engineering Section of the C.S.I.R.O. The first-stage study of the sulphiding of ilmenite is described in this paper by WHITEHEAD and RESTARICK.

To enable the partial pressure of sulphur, reaction temperature and rate of sulphur supply to be varied independently, it was decided to supply the sulphur as vapour mixed with an inert carrier gas. To achieve the required degree of control over the reaction system, a fluidised-bed reactor seemed the best solution. This also eased the materials problem: all easily available metals and many refractories are attacked by sulphur vapour, but because of its simplicity of construction, the fluidised reactor vessel could be considered expendable and was therefore made of stainless-steel tubing.

Sulphided ilmenite in a sulphur atmosphere agglomerates severely and prevents fluidisation. This can be overcome by diluting with an inert substance. Partially sulphided and leached ilmenite is non-agglomerative and is a suitable diluent to prevent agglomeration with fresh ilmenite.

The effects on the reaction of varying temperature, reaction time, sulphur feed rate, and of the presence of active carbon, carbon dioxide and water vapour are as follows. (1) The rate of sulphiding of ilmenite increases with temperature in the range 600-800°C. Above 800°, unless SO_2 is removed, the rate may be limited by back reaction. (2) The rate of sulphiding is independent of the degree of conversion until the sulphur content as FeS in the sulphided ilmenite reaches about 12 per cent. Beyond this the rate of reaction falls off and long reaction times and/or high sulphur partial pressures are required for complete conversion. During the constant-rate period, the rate of sulphiding appears to be approximately proportional to the sulphur concentration. (3) The proportion of sulphur present in the form of FeS_2 decreases with increasing temperature and reaction time. (4) The presence of active carbon increases the rate of sulphiding, mainly by reducing the SO_2 to elemental sulphur and thus suppressing the back reaction. (5) Carbon dioxide and water vapour have negligible effect on the reaction. Thus it would be feasible to heat the system directly by hot gases from the combustion of fuels.

Chlorination of Ilmenite—Practical Aspects of the Design, Construction and Operation of Small Scale Fluid Bed Reactors

by A. W. NORDIN

IN this paper the author describes some of the experimental techniques used in the development of a continuous process for producing rutile by chlorinating ilmenite. In addition to a study of the fluidising properties of the ilmenites used, reaction rate data were also obtained. The process has two reaction stages, oxidation and chlorination, both of which can be carried out in fluid bed reactors; the chemical composition of the ilmenites is given in Table I.

Among equipment were perspex models for simulating fluidising conditions in either batch or continuous reactors. One model was of 6 in. diameter and arranged with from one to five stages; it employed a discharge system of the overflow type and the solids were fed as close as possible to the air distributor of any particular bed, the distributors themselves being fitted with brass bubble caps.

Reaction equipment included an oil-fired furnace in which was situated a 3-in. two-stage silica tube reactor (Fig. 1).

The fluidisation properties obtained by observations with the models are represented graphically as plots of bed pressure drop against air flow rate, an example being Fig. 2. Observations of the quality of fluidisation are noted in this figure and the definitions of the zones are summarised in Table II.

Chlorination and Oxidation Results

Oxidation experiments which were conducted at temperatures of 1000°C indicated that the time for complete oxidation was not much affected by variation in gas flow rate, gas composition and bed aspect ratio.

Successful continuous chlorination experiments were performed in a 3-in. single-stage reactor, using pure chlorine as a fluidising gas, the bed temperature being 1100°C. Successful tests were carried out in the two-stage oil-fired equipment, with a bed aspect ratio of unity and again at bed temperature of 1100°C. The results show that reaction rate decreases with increase in chlorine utilisation (defined as the ratio of the weight of chlorine consumed to the weight of chlorine available and is expressed as a cumulative percentage). The overall chlorine utilisation (the sum of the bottom and top stage utilisations), in the case of two chlorinating stages, increased at first with the bottom stage utilisation. It ceased to rise once its maximum reached approximately 43 per cent utilisation, the predicted value for the oxidised ilmenite chlorine at 1100°C. Beyond this point, increasing the bottom stage utilisation decreased the top batch stage utilisation.

Favourable Fluidising Conditions

The reactions were best carried out in the boiling zone, as disadvantages derived from operation in many of the other zones. Thus, with channelling, large volumes of gas pass through a

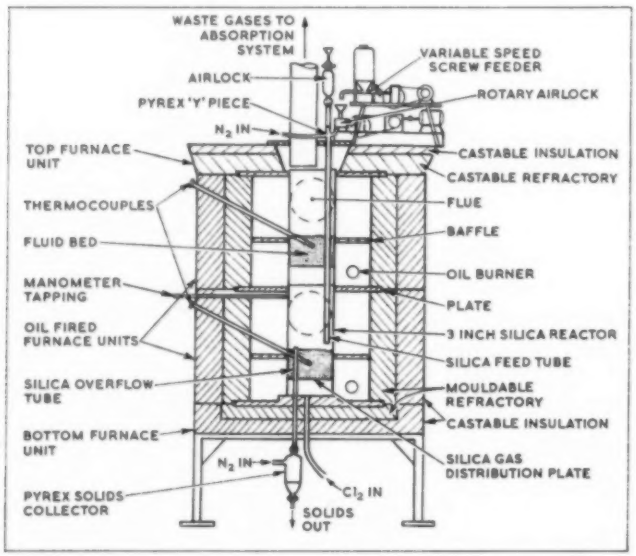

Fig. 1. Sectional diagram of oil-fired furnace showing two-stage reactor of 3-in. silica tube.

TABLE I—Chemical Composition of Ilmenite Samples

Compound	Per cent by wt.	
	Capel	Stradbroke Island
Ferrous oxide (FeO)	26.5	31.6
Ferric oxide (Fe_2O_3)	17.0	14.4
Titanium oxide (TiO_2)	53.7	50.1
Chromic oxide (Cr_2O_3)	—*	0.2
Insoluble (silicates)	0.5	0.9
	97.7	97.2

* Not assayed.

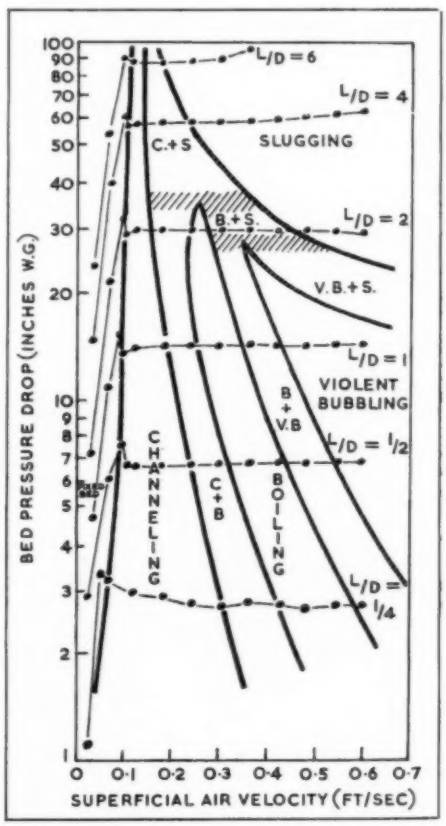

Fig. 2. One of the fluidisation quality diagrams shown by Nordin. Experiments carried out in a 6-in. model reactor with Capel ilmenite.

TABLE II—Definition of Quality of Fluidisation Zones

Zone	Definition
Fixed Bed	Bed not fluidized; gas flows between packed solid particles; as gas flow increases, bed expands to position of loosest packing, which is point of minimum fluidisation.
Channelling	Bubbles rising through gently fluidised bed; virtually no bed turnover.
Channelling + Boiling	Bubbles boiling randomly at bed surface; much of surface is quiescent; some slow bed turnover.
Boiling	All of bed surface boiling strongly; none of surface is quiescent; good bed turnover.
Boiling + Violent Bubbling	All of bed surface boiling strongly with some violent eruptions at spasmodic intervals; some solids being lifted by large bubbles breaking surface; strong bed turnover.
Violent Bubbling	All of bed surface erupting violently all of the time; solids thrown up erratically by the very large bubbles breaking surface.
Violent Bubbling + Slugging	Violent bubbling with a tendency to slug; huge bubbles tending to coalesce and lift top of bed as a slug; very violent agitation.
Slugging	Whole bed slugs from near bottom to top; bubbles grow to diameter of tube and move upwards, lifting slugs of solids in a piston-like fashion; characterised by violently varying bed pressure drop.
Channelling + Slugging	As for channelling, but a tendency to slug; bubbles plus some small slugs rising through gently fluidising bed; some bed turnover.
Boiling + Slugging	As for boiling, with a tendency to slug, particularly at top of bed; good bed turnover.

few flow paths thereby giving a non-homogeneous bed. In the violent bubbling zone the bed turnover is faster but the gas solid contact is poor. In the slugging zone operation is very irregular, the solids are reduced by attrition and the reactor materials can become unduly worn.

For chlorination the most suitable temperature is 1100°C., at which temperature there is virtually complete freedom from sintering. A suitable gas velocity, given an aspect ratio of unity, is about 0.35 ft per sec (measured at bed temperature).

Details of Apparatus

The distribution plate of the 6-in. model is noteworthy; it was provided with 19 bubble caps, each with three 0.058-in. diameter holes screwed into a ¼ in. thick perspex plate. The high temperature ruled out the use of sintered porous plates for distributors.

The original screw feeder of the oil-fired reactor was a source of difficulty but was made to work satisfactorily by fitting a rotary air-lock, consisting of a neoprene "Jabsco" pump impeller rotating in a steel case, between feeder and reactor. Other modifications found necessary on the feed system were fitting the feed tube so that it passed vertically down through the upper distribution plate to the lower stage. A slow stream of dry nitrogen was passed down the tube. These alterations were necessary to prevent solids sintering in the feed tube and also to prevent condensation of pheric chloride on the solids.

A persistent problem not completely solved at the time the paper was written was blocking of the top distributor plate. The solution contemplated was to increase the size of the distributor cap holes.

Fluid Roasting of Zinc Plant Residues at Risdon, Tasmania

by A. R. GORDON, R. W. PICKERING, O. G. WOODWARD

RESIDUES from electrolytic zinc plants contain recoverable quantities of zinc and at the Risdon plant, where the work described was carried out, a typical residue would contain about 23 per cent zinc and 31 per cent iron as oxide.

A typical sieve analysis of roaster feed is:

+14 mesh Tyler	0
−14 +28 mesh	14
−28 +48 mesh	25
−48 +65 mesh	12
−65 +100 mesh	13
−100 mesh	36

and the bulk density is 84 lb/cu. ft.

The residues are treated with sulphuric acid and the product roasted in order to render the zinc content soluble and the ferric content insoluble, so that the separation and recovery of zinc are facilitated.

Hitherto the important roasting operation, which requires heat, has been performed in moving-bed shaft furnaces, hearth furnaces and rotary kilns; however, the authors of this paper investigated the suitability of a fluid bed process for roasting.

They describe a number of experiments conducted with 6 in. and 24 in. diameter fluid bed roasters, which gave them perhaps limited guidance for the design of a projected 10 ft diameter roaster and for suitable operating conditions. The results of many experiments showed that good fluidising and good metallurgical results in terms of high zinc solubility did not necessarily go hand in hand. Of internal fuels, coal, iron and acetylene were tried, and oil proved to be the most satisfactory; coal was difficult to distribute uniformly throughout the fluid bed. Bubble caps were the preferred means of distributing the fluidising gas, and it seemed that the oil fuel should be distributed by means of tubes which terminated just above each bubble cap.

Other conclusions were that the optimum fuel burning rate is limited by the available oxygen and experiments with enriched air proved worthy of repetition on the plant scale.

In the above tests, the fluidising air flow was 8.5 cfm per sq ft. The feed rate was usually 45 lb/(hr) (ft²), except in the case of some runs with oxygen, in which the rate was 150 lb/(hr) (ft²), these rates being approximately equivalent to rates of 37 and 125 tons per day for the 10 ft roaster.

Plant Roasters

Details of the 10 ft diameter roaster, designed on the basis of information obtained from the 2 ft roaster, are given in Fig. 3. Solid was fed by means of a screw feeder discharging into an inclined 6 in. diameter stainless-steel feed pipe passing through the wall of the roaster. This tube was purged continuously with a flow of air. The material left the roaster through a stainless-steel overflow pipe, the original overflow level being 6 ft. This pipe discharged into a vertical leg connected with a quench cone.

Other features of the roaster were the 19 ft of "freeboard", twin stainless-steel cyclones in series for the dust-laden exit gases and, on the instrumentation side, a flue gas analyser based upon the paramagnetic properties of the gas. Considerable attention to the gas sampling system was necessary to ensure a clean reliable sample. The fluidising air was distributed by a plate with bubble caps arranged on 14 in. equilateral triangular pitch.

Experiences with 10 ft Roaster

When the first attempts were made to burn oil in the fluidised bed a number of difficulties rapidly developed; most of the oil tubes became blocked, the cyclones frequently choked and, most serious of all, the roaster shell burned through.

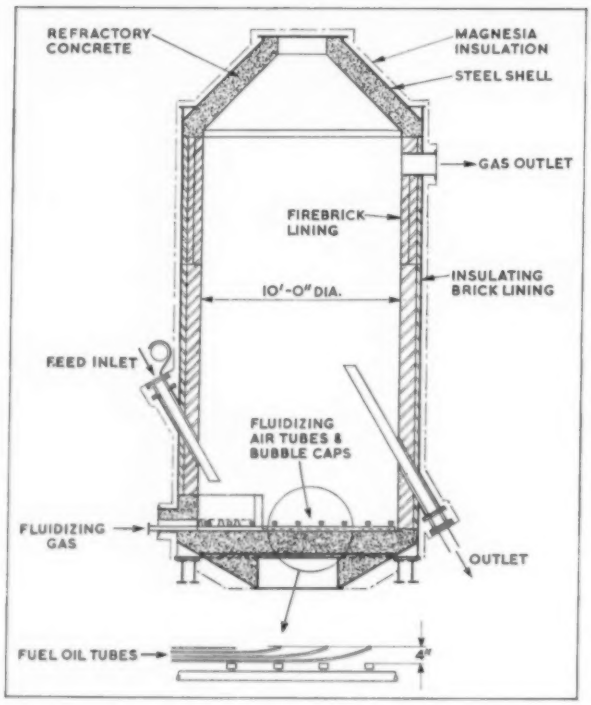

Fig. 3. Diagram of fluid bed roaster with detail of fuel oil tubes.

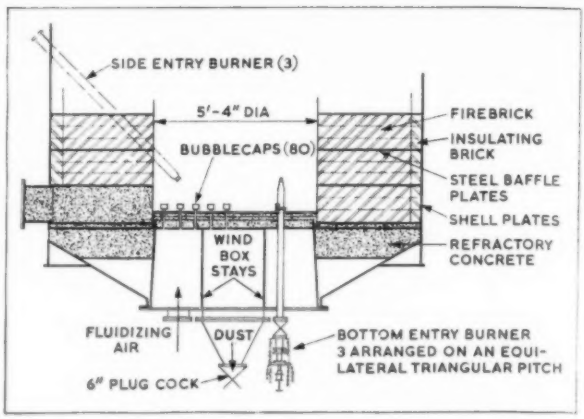

Fig. 5. Reduced diameter fluid bed roaster, with retractable oil burner.

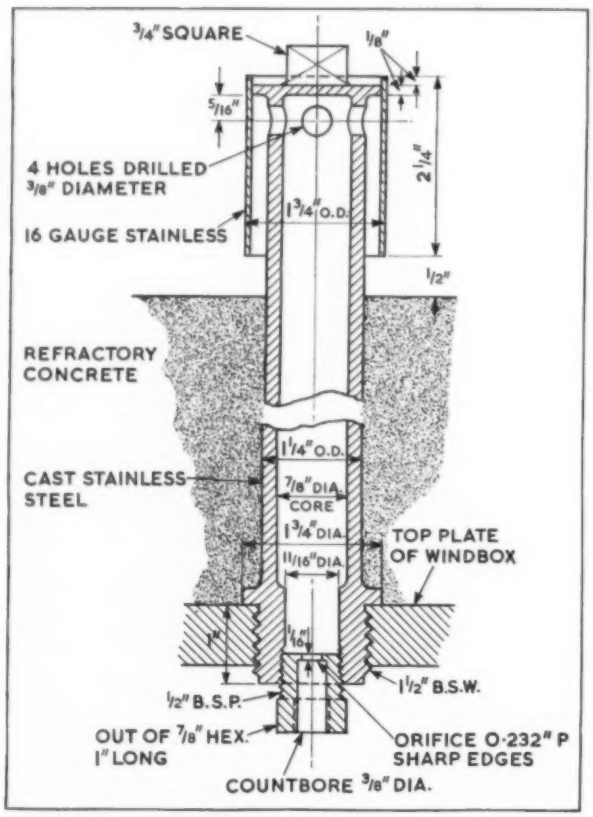

Fig. 4. Design of bubble cap for distributing fluidising gas.

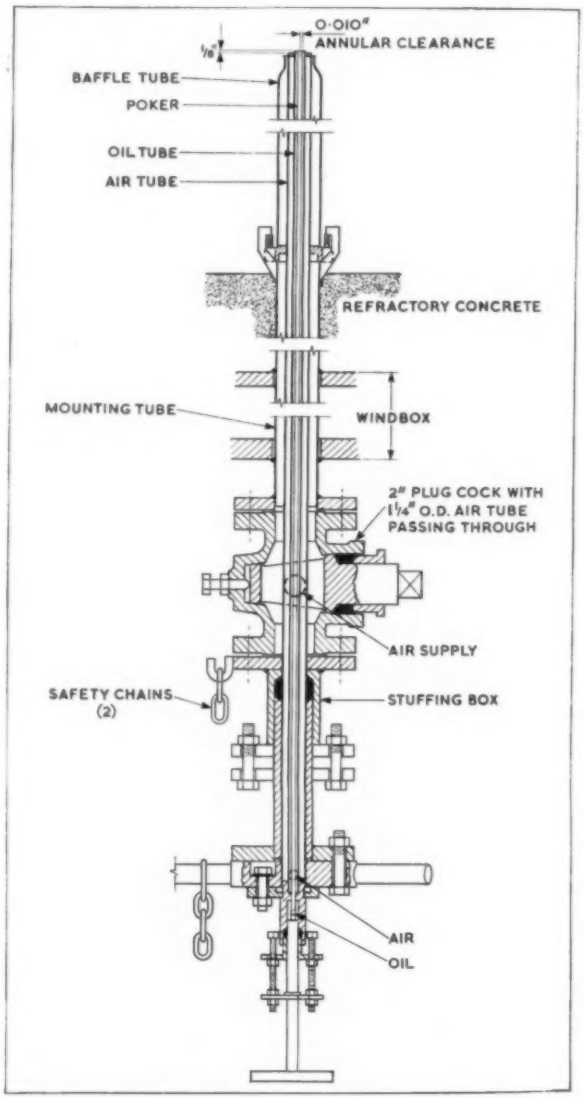

Fig. 6. Details of successful retractable oil burner.

Examination revealed a situation as discouraging as it was dramatic; considerable agglomeration of the bed had occurred and the set material had been reduced to a hard, dense mass some 6 ft in diameter and 2 ft in thickness and nearly all the oil tubes were burnt off about 2 ft from their point of entry to the roaster. The major cause of this setback was considered to be inadequate fluidisation. The damage to the shell was caused by the burning of oil between lining and itself.

The situation called for alterations to the roaster and some of its ancillary equipment. Thus, to improve fluidising conditions, a sliding-vane positive-displacement blower was installed which was capable of giving an air supply at higher pressure than previously. The orifices beneath the bubble caps were reduced in size to increase the pressure drop across them. In addition, new burners were employed of a type successfully tested in the 2 ft roaster. These changes led to a marked improvement in performance, but sintering still occurred when attempts were made to burn oil.

Further detailed improvements were to prevent air by-passing through the refractory walls and to reline the furnace to a height of about 8 ft. The operations which followed were physically satisfactory but metallurgically indifferent; the bed temperatures were in the range 760-610°C and bed heights were 6-8 ft. the latter height giving the better performance.

The Use of Oxygen

After making modification of the bubble caps to prevent run-back—their skirts were lengthened—and the fitting of a non-return valve in the fluidising air line—both measures made necessary by the presence of a Roots type blower (installed for the oxygen operations)—the investigators carried out a series of experiments with enriched air; a marked improvement in operation was noted. The amount of oxygen varied between 100 and 400 cfm in a total fluidising gas flow of 1120 cfm, but there appeared to be no advantage in operating with oxygen proportions greater than the lower figure.

Roaster with Reduced Diameter

The diameter of the 10 ft roaster was reduced (by additional internal brickwork) to 5 ft 4 in., in order to determine its performance at high specific feed rates. Experience with enriched air showed that increasing the fluidising gas flow from 14.1 cfm/sq ft to 18.25 cfm/sq ft and increasing bed temperature from 610°C to 650°C at high feed rates gave good results, such as a lower content of soluble iron. As with the 10 ft column, operating results obtained with the reduced diameter were presented in tabular form by the authors.

The authors' discussion of experiences and of results obtained, embraced also considerations affecting the reaction.

Among the more serious troubles encountered with the modified roaster was a tendency for the bed material to run back into the fluidising tubes.

Burner design was another problem and many arrangements were tried. The most successful appeared to be the one shown in Fig. 6. This has an air jacket around the central oil tube, whereas the oil tube itself has inserted within it a movable rod, which prevents particles from blocking the tube and can be used to clear blockages. The most satisfactory type of burner can be extracted from the roaster through a plug cock, and should terminate 1 ft above the bubble cap. However, this arrangement, satisfactory on the 5 ft 4 in. roaster, had still to be tried on the larger size roaster.

For starting up, the best procedure was to heat the bed by means of an oil burner firing through a port in the wall above the bed. By this means a bed temperature of 600°C could be reached in about 35 hr.

Gas cleaning equipment for the 10 ft roaster consisted originally of two cyclones; a number of caking troubles occurred in them although these were reduced by an improvement in the quality of the dust collected. Bridging at the outlets of the cyclones also took place and was overcome by practically doubling their diameters.

Future Work

In a redesigned 10 ft roaster it is planned to operate with a total gas flow rate of 19.4 cfm/ft^2. On the basis of a heat requirement of 490,000 CHU/ton of feed, the authors give figures for oil consumption, feed rate, etc., at different degrees of enrichment. For 55 per cent O_2 in the fluidising gas, the feed rate estimated is 275 ton/day and for 100 per cent O_2, 550 ton/day.

The Influence of Solid Characteristics on the Performance of Fluidised-bed Reactors for Non-catalytic Operations

by A. B. WHITEHEAD

AGGLOMERATION during processing is one of the chief obstacles to the more universal application of the fluidised-bed technique. Even slight agglomeration, which may produce a lightly sintered bed when the reactor is shut down, may prevent complete refluidisation of the bed after a long shut down and severe agglomeration can make a fluidised-bed system inoperable. On the other hand, the deliberate promotion of a controlled degree of agglomeration, e.g., to produce desired particle sizes, or to allow the use of higher space velocities, has many potential applications in the chemical and metallurgical industries.

Factors affecting agglomeration are:

(1) Particle size—the smaller the particle being fluidised, the greater the difficulties with agglomeration.

(2) Space velocity—with increased velocity, particle-to-particle contact is of shorter duration, and the concentration of solids in the bed is much reduced, thus reducing the probability of agglomeration.

(3) Changes in the proportion of particle surface capable of bonding—influenced by the chemical and physical composition of the bed; for instance, the presence of an inert diluent.

(4) Bed temperature—usually the most critical factor; generally, the higher the temperature, the greater the chance of agglomeration. Sintering tendencies may start several hundred degrees below the usually reported melting point.

Although high space velocities can assist in controlling agglomeration, they may bring other disadvantages: incomplete reaction with loss of product quality; reduction of effective operating range of a unit; adverse effect on gas-solid contact; loss of temperature control of the reactor arising from reaction occurring in the freeboard which is less closely controllable than the fluidised bed.

Dilution with inert material has been very successfully used to make possible studies of gas-solid reactions with pronounced agglomerative studies. In commercial applications, addition of inerts has limitations, especially in endothermic processes, although in some special instances the diluent may be a convenient means of supplying heat to the bed, either acting as a fuel or being preheated. There may also be difficulties in separating the diluent after the fluidised-bed stage.

Another possibility for treating an otherwise severely agglomerating material is to make a large proportion of the bed particles inert by coating with a non-agglomerative powder or by chemical change.

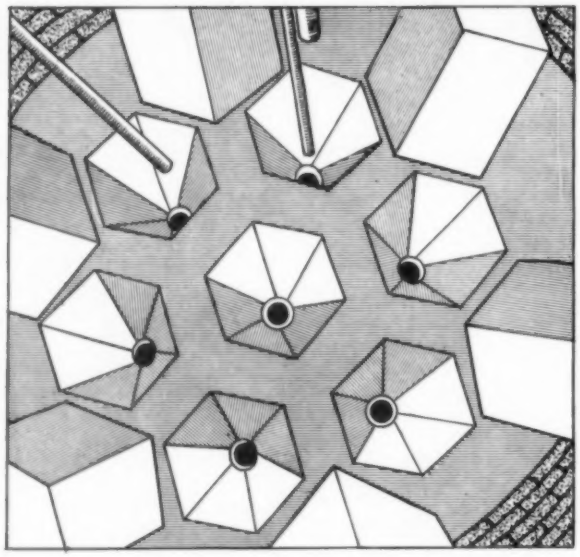

Fig. 7. A fluidising gas distributor designed to reduce agglomeration, consisting of a series of interlocking cones.

In the laboratory it may be possible to choose a coarse particle size to avoid agglomeration. In practice, it would rarely be possible to discard the fines.

Another proposal is to draw off large agglomerate accumulations from the base of the fluidised bed but this would seem to need the provision of a number of bottom outlets.

The use of a series of interlocking cones as a reactor base, as shown in the illustration, has proved completely effective in preventing the formation of agglomerates under conditions that would normally be severely agglomerating, due to an increasing velocity distribution towards the base and highly turbulent conditions in the cones. This design also eliminates stagnant regions and prevents deposits forming on the reactor walls.

Operating temperature is usually a compromise between the desire for a fast reaction, the need for a certain product composition and the necessity to keep the system operable.

When units are defluidised, the whole bed may tend to sinter together. To avoid trouble during planned shut-downs, the bed can be periodically refluidised for a few seconds; or prior cooling under fluidising will prevent sintering while the bed is stagnant; or in some cases the bed composition can be changed by temperature adjustment before shutting down; or an inert diluent can be added. In the case of unforeseen shut-downs, if the main blowers are inoperative, a small auxiliary blower could be used to refluidise the bed. When using the multi-cone distributor illustrated, the application of high-pressure air to each cone in turn will refluidise a lightly sintered bed.

It is difficult to predict full-scale behaviour with agglomerating systems from results in small-scale reactors and, therefore, laboratory investigations should be carried out in the largest practicable units possible. Batch operation can give very misleading indications.

Methods for detecting agglomeration include (1) physical exploration with a probe; (2) simultaneous temperature measurements at a number of points in the bed—any sudden temperature divergence is a sign of stagnation; (3) measuring pressure differential across the bed—as the bed agglomerates, there is a gradual reduction in the frequency of the pressure oscillations; (4) visual observation—this can be deceptive, as a bed usually defluidises from the bottom upwards; (5) measurement of gas distribution by orifice meters or rotameters will rapidly indicate stagnant regions or channels.

Fluidised Bed Roasting of Copper Concentrates

by A. B. WHITEHEAD and R. W. URIE

As part of a commercially sponsored programme for the technical evaluation of the hydrometallurgical recovery of copper from Australian chalcopyrite concentrates, the Chemical Engineering Section of C.S.I.R.O. have been investigating the fluidised bed roasting of copper concentrates. To obtain the desired leaching characteristics in the calcines, the object was to control roasting to produce copper oxide rather than copper sulphate. Two series of experiments were carried out on copper concentrates from the four major Australian copper mines—Mt. Morgan, Mt. Lyell, Mt. Isa and Peko; first, in a large 3 ft 6 in. diameter slurry-fed pilot plant roaster operated under autogenous conditions and subsequently in a 12 in. diameter ex-

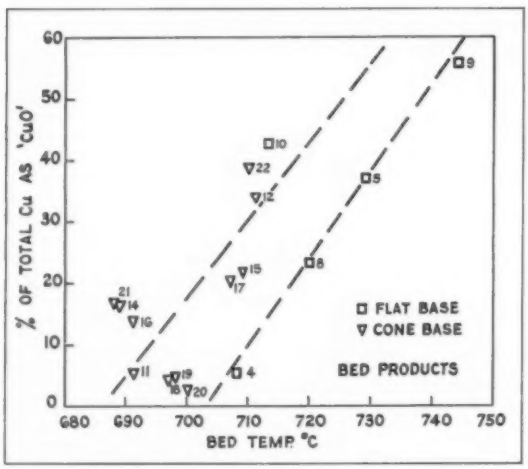

Fig. 8. The effect of temperature on a conversion of copper to "CuO" in a fluidised bed.

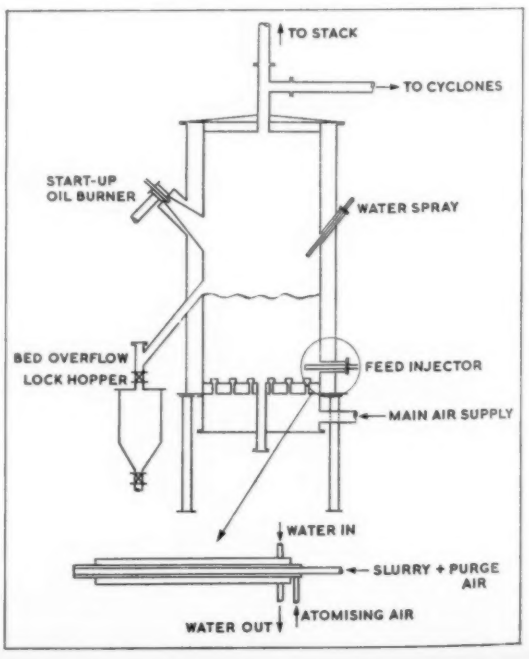

Fig. 9. Diagram of roaster for copper concentrates, and slurry feeding ejector.

ternally-heated roaster provided with a dry feed system.

The objective of producing consistently calcines with the required leaching properties was successfully achieved. The large slurry-fed fluidised roaster, in which temperature was controlled by water injection, proved simple and inherently stable in operation. Agglomeration of the calcines, a potential source of trouble, could be controlled by operating with a high fluid velocity. A multicone distributor developed during the investigation, by providing for a continuous increase in fluidising velocity towards the bottom of the bed, was most effective in controlling agglomeration over a wide range of velocities.

For the control of calcine composition, bed temperature is the most important variable, the optimum lying in the range 690-720°C, depending on the particular concentrate and other operating conditions. The close control of temperature necessary to give the desired calcine composition can be achieved with relatively simple equipment and instrumentation.

Up to 50 per cent of the copper in the calcines may be converted to CuO before the copper leach efficiency is seriously reduced due to copper ferrite formation. Increase in CuO content is usually accompanied by a fall in the extraction of iron.

Spent electrolyte can be used instead of water for feed slurry preparation and temperature control in the roaster, but it seems unlikely that more than one-third of the total available quantity could be disposed of in this way.

In the case of concentrates with gold and silver contents, no significant loss of gold by volatilisation occurs during roasting and recovery of gold from leached residues by cyanidation is excellent; recovery of silver was less satisfactory.

Some Problems Affecting the Carbonisation of Coal in a Fluidised Bed

by K. McG. BOWLING and P. L. WATERS

THE heat necessary for the carbonisation of coal (about 400 Btu/lb at 500°C) is much more rapidly, efficiently and uniformly transferred to coal in a fluidised bed than in a fixed bed. The main practical difficulties to be overcome are (1) the possibility of blockages in the by-product recovery system and contamination of liquid by-products by the entrainment of fine dust, and (2) solidification of the bed by agglomeration of softened coal particles.

Experiments have been carried out in a 2 in. diameter column at the Division of Coal Research, C.S.I.R.O., to determine the influence of elutriation time, gas velocity and bed depth on losses of fines by elutriation from coal. With a bed depth of 8¾ in., air velocity 0.28 ft/sec, losses were highest when very small particles were present. Losses could be cut by about two-thirds by removing the −120 B.S. mesh fraction before fluidising, and appreciably reduced by removing only the −200 B.S. mesh particles. For the size ranges tested, doubling the gas velocity almost trebles the losses. Losses per unit weight of bed are high with shallow beds but are reduced inversely as bed depth increases. It follows that the total elutriation loss from a fluidised bed is roughly constant irrespective of the depth of the bed.

In the 50 lb/hr experimental carbonising plant, developed by the authors, the problems arising from the entrainment of fine dust were overcome, first by a pre-treatment stage, in which the ultra-fine particles from the crushed coal were removed by air-elutriation prior to carbonisation, and finally by removing particles entrained in the gas stream leaving the carboniser in two high-efficiency cyclones kept sufficiently hot to prevent pitch deposition.

Partial or complete solidification of the fluidised bed may result from the coking of coal. Coking troubles may be avoided by (1) using non-caking coals, (2) reducing caking power by pre-oxidation—which will, however, also reduce the tar yield—or (3) adding suitable quantities of an inert material such as char. This latter measure has been adopted by C.S.I.R.O. and trouble-free operation with Liddell coal has been obtained at char:coal ratios of about 5:1. The caking power of coals can also be reduced by adding certain chemicals, e.g., boric acid. The method of feeding coal into the carboniser should provide for rapid dispersion of the coal through the bed; the most popular method has been to convey the coal pneumatically in the fluidising gas.

In a commercial carbonising plant, if air is used as the fluidising medium and the coal:air ratio is suitably adjusted, partial combustion of the fluidised char will supply the heat necessary for the carbonisation process which is endothermic. Alternatively, conditions can be adjusted to provide for preferential burning of the tar rather than of the char.

Low-temperature tars produced by the fluidised-bed process demand different processing from that appropriate to high-temperature tars. There are no existing markets for the likely products. The commercial future for the fluidised-bed carbonisation of coal therefore poses economic as well as technical problems.

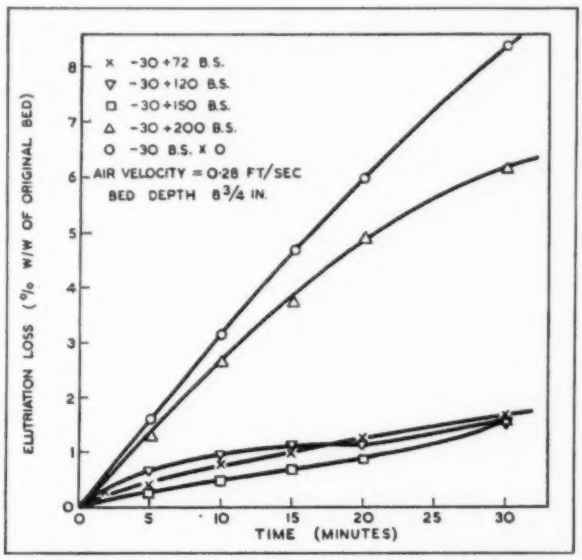

Fig. 10. Elutriation losses increase with residence time in bed and are highest when very small particles are present.

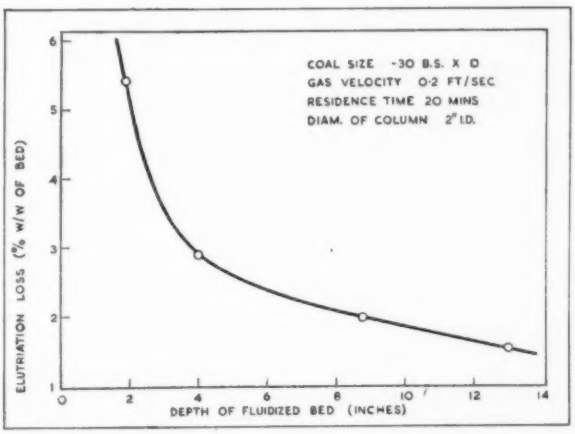

Fig. 11. Effect of bed depth upon loss of fine particles by elutriation.

Abstracts

GROUP A — FLUID DYNAMICS

Fundamental Aspects of Solids-Gas Flow. Part V: The Effects of Fluid Turbulence on the Particle Drag Coefficient*

This article presents an analysis of the experimental evidence presently available on the effects of the free-stream turbulence on particulate momentum transfer, obtained from both wind-tunnel tests with stationary bodies and from single-particle systems in which the solid is moving freely with the stream. Original experimental measurements recently obtained with a radio-tracer technique are also included. The drag coefficient appears to depend primarily on the magnitude of the relative turbulence intensity and on the particle Reynolds number, while acceleration has negligible effects. Increasing intensities cause a systematic regression of the transition region of the drag coefficient curve towards lower Reynolds numbers, together with a moderate increase of the drag coefficients for both the sub-critical and supercritical Reynolds numbers. The momentum transfer behaviour suggests the occurrence of a laminar-turbulent transition in the attached boundary layer at a critical value of the Reynolds number for a given relative intensity. Indirect evidence involving heat and mass transfer phenomena appears to support this hypothesis.

L. B. Torobin and W. H. Gauvin, *Can. J. Chem. Eng.*, 1960, **6**, 189.

A Study of Aggregative Fluidisation

The effects of size distribution on the expansion of a gas-solid fluidised system have been studied with the fluidisation of glass beads and char particles by air in a 2¼-in.-diameter column.

Log-probability distributions of 5, 10, 25 and 100-fold in size variation, with an average size of 271 microns, were tested and changes in the average diameter of the bed due to elutriation were obtained.

Systematic tests were also carried out with beds of closely sized particles ranging from 74 to 912 microns in size.

The results show that size distribution has no effect on the shape of the expansion curve above the point of good fluidisation and below the point of elutriation, for beds of the same average diameter as defined by the log probability scale. Various correlations have been tested, but none has proved satisfactory for aggregative fluidisation.

G. Davies and D. B. Robinson, *Can. J. Chem. Eng.*, 1960, **6**, 175.

Fundamental Aspects of Solids-Gas Flow. Part IV: The Effects of Particle Rotation, Roughness and Shape

An examination of the effect of rotation, surface roughness and shape on the motion and drag of single particles entrained in a fluid. Rotation appears to influence the drag coefficient at low Reynolds numbers but slightly affects the linearity of the motion and may thus be of significance in particle entrainment in conveyed solids-gas systems. Negative lift observed in the critical Reynolds number region occurs through asymmetry in the wake structure. Surface roughness produces an early transition to a turbulent boundary layer at higher Re values, resulting in an appreciable decrease in drag coefficient, but it may increase the drag at low Reynolds numbers.

The effect of particle shape is more complex than previously published analyses have suggested. The available empirical correlations are discussed and compared, and indicate a dependency on the particle-to-fluid density ratio.

L. B. Torobin and W. H. Gauvin, *Can. J. Chem. Eng.*, 1960, **5**, 142.

Energy Losses in a Compressible Emulsion in Vertical Motion

The author examines the losses in a compression column containing an air and water emulsion. This leads him to the theoretical study of coalescence which causes the instability of homogeneous emulsions.

The experimental results obtained on two installations, differing greatly from one another, confirm the theoretically predicted data regarding the losses due to slipping and other important parameters.

J. Bonnin, *Gen. Chim.*, S.1960-6-210.

* Author's abstract.

Maximum Liquid Transport. Flow of Gas-liquid Mixtures in Vertical Tubes

Relationships between pipe length, diameter and material characteristics are used to calculate maximum liquid transport in vertical tubes for the same amount of liquid transport. For the same amount of liquid transport, an increase in the amount of gas in a gas-liquid mixture decreases the specific weight and increases the frictional losses. For a given pressure drop, the fluid transport through the introduction of gas can only be increased up to a certain limit. For long pipes, the maximum liquid transport depends on the fourth power of the available pressure drop; in short pipes, it depends on the 3/2 power.

A. C. Nemet, *Ind. Eng. Chem.*, 1961, **33**, (2), 151.

Concentration Distribution in Two-phase Pipe Flow

An investigation of the concentration distribution of solid particles in two-phase turbulent pipe-flow, in the case of small loading, where gravity effect is small. The authors show that, through their inertia, solid particles transported across the velocity field of the turbulent stream by turbulent diffusion are decelerated in the region near the wall and those transported toward the high velocity core are accelerated. In the core, the particles have a lower mean velocity than the stream, while near the wall the mean particle velocity is higher. This gives rise to a non-uniform distribution of solid particles in fully developed turbulent pipe flow. Experimental determination of concentration distribution was made with an impact disc particle counter system.

S. L. Soo and J. A. Regalbuto, *Can. J. Chem.*, 1960, **5**, 160.

Mixing in a Jet-stirred Reactor*

Measurements were made of the overall mixing in a gas phase flow reactor stirred by the entering feed jets. The mixing was studied by following the decrease in exit concentration after sharp cut-off of a radioactive tracer gas, krypton-85. The data showed that over the entire range of average residence times investigated, about 0.4 to 16 sec, the reactor behaved as though 85 per cent of its volume was perfectly stirred and the remaining 15 per cent was in piston flow in series with the stirred region. Relocation of the feed jets would probably increase the fraction of volume that is effectively perfectly stirred to about 95 per cent.

William Bartok, Carl E. Heath and Malcolm A. Weiss, *A.I.Ch.E.J.*, 1960, **6** (4), 685.

Pressure Drop of Single Phase Flow Through Raschig Ring Type Tower Packings: Effect of Hole Size

The paper deals with the effects of hole size, of cylindrical tower packing, on the pressure drop of single-phase flow. The packing used was ¾ in. O.D. and ¾ in. high with hole diameters 0, 0.197 and 0.394 in. respectively.

The hole size had a profound effect on the correlation of pressure drop data. This was attributed to the characteristics of the spaces inside and outside the packing units. The pressure drop is greatest for the solid block packing and least for the larger hole packing. Experimental data for the solid block packing can be correlated by means of Ergun's equation.

Liang-Tseng Fan, *Can. J. Chem. Eng.*, 1960, **5**, 138.

Effects of Solids on Turbulence in a Fluid*

The effect of solid particles on fluid turbulence was studied for fully developed flows of slurries in a vertical 3-in. pipe for solids concentrations ranging from 0.13 to 2.5 volume per cent. Point source turbulent diffusion data in the slurry flows were compared with data for flows without solids present. The solids do not appear to have a large effect on the diffusion rate unless there is an appreciable average slip velocity between the solids and the fluid and unless the solids concentration is high enough.

Hisao Kada and Thomas J. Hanratty, *A.I.Ch.E.J.*, 1960, **6** (4), 624.

Brit. Chem. Eng. Abstracts: **FOLIO No. 1**

GROUP B — HEAT TRANSFER

Evaporation of Stationary Droplets in High-temperature Surroundings

The results of a study of the rate of evaporation of stationary droplets of water, methanol, cumene, pentane and benzene, carried out in an electrically heated 9-in. stainless-steel sphere are presented. The gaseous environment consisted of the superheated vapour of the liquid under investigation; drop diameters ranged from 0.4 to 1.4 mm, temperatures from 100°C to 550°C and wall and gas temperatures were, in all cases, the same.

The evaporation rate of stationary droplets under these conditions does not appear to be governed by the rate of heat transfer. Attempts to correlate results on the basis of Godsave's model or on Spalding's approach were unsuccessful.

Evaporation rates appear to be predicted satisfactorily by the expression:

$$\left[\frac{dm}{d\theta}\frac{Cp}{ndk_f}\right]Pr^{-\frac{1}{3}} = 3.2\,B^{0.97}$$

over the range $0.03 < B < 1.0$ where B is Spalding's transfer number.

This correlation indicates that the mechanism of this evaporation is governed by the effect of the evolved vapours on the boundary layer flow.

T. W. Hoffman and W. H. Gauvin, *Can. J. Chem. Eng.*, 1960, **5**, 129.

Design of Direct-contact Cooler-condensers

A method for sizing direct-contact cooler-condensers is presented which avoids many oversimplifications of earlier techniques. The mass transfer characteristics are represented by a constant height of a gas phase mass transfer unit, obtainable from the plentiful data on simple absorption. A suitable analogy provided corresponding heat-transfer coefficients.

The design method, when applied to several typical condensation operations, shows that many systems tend to become supersaturated. The liquid phase heat transfer resistance was found to be negligible. At high humidity levels the enthalpy potential method deviated considerably from the exact solution. At low humidity levels, the agreement was satisfactory only for systems where Prandtl-to-Schmidt ratio approached unity.

D. R. Olander, *Ind. Eng. Chem.*, 1961, **53** (2), 121.

Use a "Z" Factor to Measure Thermal Performance*

The Z factor suggested here is proportional to the inverse of the overall heat-transfer coefficient:

$$Z = \Delta T_{LM}/\Delta t$$

for no phase change, or

$$Z = \Delta T_{LM}/\left(\Delta t + \frac{\Delta H}{Cp}\right)$$

for complete phase change. ΔT_{LM} = corrected log mean temp. difference; Δt = temp. change across tube or shell of exchanger; ΔH = latent heat of vaporisation; Cp = heat capacity. Z is calculated in terms of flow rates for the cases of no phase change and complete phase change.

E. I. Motte, *Petrol. Engr.*, 1960, **32** (6), C47.

Increasing Evaporator Capacity with Polished Tubes*

Replacement of pickled-finish stainless-steel tubes with polished-bore tubes in a forced-circulation evaporator increased capacity about 80 per cent in a tomato-paste concentration operation. The change to polished tubes decreased apparent heat damage to the product and decreased tube fouling from burned-on material. Overall heat-transfer coefficients indicated that polished bore tubes may be operated for a considerably longer time than unpolished tubes before it is necessary to shut down for clean-out.

P. W. Kilpatrick and E. Breitwieser, *Ind. Eng. Chem.*, 1961, **53**, 2, 119.

These Four Problems Guide Cooling-tower Design*

Four problems which are being used as the criteria for the latest designs of cooling towers—structural and mechanical equipment failures, thermal performance deficiencies and fungal attack on redwood—are discussed.

R. W. Maze, *Oil Gas J.*, 1960, **58** (31), 122-8.

Now Consider Evaporative Cooling

This paper describes evaporative coolers which, in a number of applications, give greater heat transfer than air coolers by offering a lower temperature heat sink. Evaporative coolers can also have advantages over a design requiring a cooling tower.

P. D. Berkeley, *Pet. Ref.*, 1961, **1**, 165.

Rate Exchangers This Computer Way

A digital computer programme is proposed to eliminate the tedious, repetitive labour from heat exchanger design. This allows the designer more time for design evaluation.

D. L. Whitley and E. E. Ludwig, *Pet. Ref.*, 1961, **1**, 147.

How to Measure Tube-wall Temperature

Metal-sheathed, oxide-insulated thermocouples, when installed correctly, measure the tube-wall temperature of fired heaters most reliably. The design of thermocouple and correct installation procedure are given. It is considered that, in the future, a method of optical scanning of the entire tube surface would be preferable to spot readings obtained with thermocouples.

F. S. Becker and F. M. Kepler, *Oil Gas J.*, 1960, **58** (29), 100-1.

GROUP C — MASS TRANSFER

The Effect of Plate Wetting Characteristics on Pulse Column Extraction Efficiency*

Liquid-liquid extraction of acetic acid from the methyl isobutyl ketone-water system was studied as a function of plate wetting characteristics and other variables in a pulsed perforated-plate extraction column.

Various combinations of stainless steel plates and polyethylene plates were used with different directions of solute transfer at a constant throughput of 82.6 lb total flow/(min) (sq ft column area) and with other operating variables in the following range:

Frequency	...	...	16 to 117.1 c./min.
Amplitude	...	...	0.492 and 0.973 in.
W/K flow ratio		...	0.46 to 2.8 lb water/lb ketone

The plate wetting characteristics were found to affect the column extraction efficiency when the solute transfer was from the continuous ketone phase to water. An all-polyethylene plate arrangement provided the best efficiency ($H.T.U._{oc} = 4.1$ in.) while an all-stainless-steel plate arrangement was less efficient ($H.T.U._{oc} = 6.1$ in.) under the most favourable operating conditions. A combination of these two arrangements in the column produced efficiencies midway between the all-plastic plate arrangement and the all-stainless-steel plate arrangement.

Within the column flooding limits the extraction efficiency did not seem to be effected by the plate wetting characteristics when the solute transfer was from water to the continuous ketone phase.

R. H. Sobotik and D. M. Himmelblau, *A.I.Ch.E.J.*, 1960, **6** (4), 619.

Axial Diffusion of Liquids in Packed Beds and End Effects*

Experimental data were obtained on the effect of bed length of packed spheres on axial diffusion coefficients with the frequency response technique used. Lengths of packed section of 6 to 174 cm were used. The end effects were eliminated by using a novel experimental technique for analyses of the inlet and outlet streams. No effects of length on D_L were found.

When the end effects were artificially introduced by using void analytical sections at the ends, then large effects of length on D_L were found. These void sections were the cause of length effects found by other experimenters. The D_L drops as the length of bed is increased. Above about 65 cm length the D_L remains constant with increase in length. The overall results are in general agreement with the data of others.

A. W. Liles and C. J. Geankoplis, *A.I.Ch.E.J.*, 1960, **6** (4), 591.

Problems of Convective Mass Transport in Electrolysis

The processes of mass transport (electrical migration, diffusion, convection) in electrolysis also led to concentration differences in the vicinity of the electrode surface and in the interior of the solution. These processes determine the magnitude of the limiting current which is a measure of the maximum velocity with which an electrochemical process can be carried out under given agitation conditions.

Examples indicate that these data can be estimated, for particularly simple cases, using dimensional analysis and the boundary layer theory from hydrodynamics.

N. Ibl, *Chem. Ing. Tech.*, 1961, **33** (2), 69.

Temperature Dependence of the Binary Diffusion Coefficient of Gases

The variation of the binary gas diffusion coefficient with temperature for the gas systems N_2-H_2 and NH_3-H_2 over the range 0-300°C.

The data were obtained by a simple and accurate steady-state flow method.

Experimental data are in agreement with equations for the prediction of gas diffusion coefficients. Correlation between data and equations is best for nearly radial systems such as N_2-H_2, rather than for systems having highly polar molecules such as NH_3.

D. S. Scott and K. E. Cox, *Can. J. Chem. Eng.*, 1960, **6**, 201.

[*Abstracts are continued on pages* 270 *and* 272]

Brit. Chem. Eng. Abstracts: **FOLIO No. 2**

Book Reviews

Annual Review in Automatic Programming, Vol. 1
Edited by R. Goodman, M.A., B.Sc.
Pergamon Press Ltd., 300 pp., 63s.

AS a result of a conference on "Automatic Programming of Digital Computers" held at Brighton Technical College on April 1, 2 and 3, 1959, it was decided to establish an Automatic Programming Information Centre. This Centre came into being on January 1, 1960. One of its aims is to produce an annual review on automatic programming, and the volume above is the first such review.

The main object of this book is to reproduce the papers read at the Brighton Conference. They have been modified so as to take into account any important points arising in the discussions. There are 18 papers in all, including an introduction by DR. A. D. BOOTH. For good measure there are also four appendices dealing with:

(1) Two papers on the logical theory of computing machines written some 25 years ago by A. M. TURING.
(2) A preliminary report of a joint German-American committee on an international algebraic language.
(3) A short bibliography on automatic programming.
(4) A list of participants in the Conference.

The introductory paper by BOOTH, which is a combination of his opening and concluding remarks to the Conference, deals with the necessity for automatic programming, its advantages and disadvantages, and with what he calls "the present muddle in the field of automatic programming".

The only point on which he sees eye to eye with the other authors, and they with each other, is that most computer users are "nitwits" and do not know what they really want.

In BOOTH'S opinion, the statement "with automatic programming time may be saved in the actual programming" is false, both in the scientific and in the commercial field. He argues that most of the work is done in numerical analysis before the problem is put on the machine.

The reviewer believes, however, that in many engineering problems (both chemical and mechanical) which are at present calculated by hand this is not the case, and automatic programming saves considerable time. This is certainly so in many routine design calculations such as orifice plate design, tube plate design, flange design, etc.

The bulk of the other papers describe specific automatic programming techniques such as PEGASUS Autocode, MERCURY Autocode, various codes for DEUCE, ZEBRA simple code, I.B.M. FLOW-MATIC, MATH-MATIC, and so on. The treatment is not simple and the book could not be recommended as a means of learning automatic programming. The manufacturers' literature is much better for this purpose.

The remaining few articles are of a general nature and cover topics such as future trends in the subject, general philosophy of automatic programming, operational experience, universal automatic codes, terminology, etc. One author, looking into the future, envisages a coding scheme which might, for example, print out: "I propose to use the Runge-Kutta method with an interval of 0.01 in x. Will this be O.K.?" It would be much more useful, however, if the machine answered this question instead of asking it.

A noticeable point on reading this book is the number of strange new words one encounters. Thus the beginner to the subject must almost have to learn a new language. The following sentence serves to illustrate this point: "The SOS debugging system provides a system of macro operations for obtaining snapshot information." At the other extreme the book abounds with rather childish words and phrases that could well have been omitted. Thus we have "programming for nitwits", "yours is a load of tripe", "simple enough for clots to understand", and so on.

In conclusion, it could not really be said that this is a book that every chemical engineer must have. It is rather a book that deserves to be found in the computer section of the chemical engineering library for reference purposes.

C. STOREY

Laboratory Handbook of Toxic Agents
by C. H. Gray, Editor-in-chief
The Royal Institute of Chemistry, London, 1960. 20s., including postage

WHEN help is needed following a laboratory accident, it is needed quickly. For this reason, it would be sensible not only to have this valuable book available, but to urge staff to read it as soon as it arrives and become familiar with its contents and layout.

The quick-reference section listing alphabetically poisonous and corrosive materials is on pale blue paper so that it can readily be found. Preceding it are an introductory chapter on general principles by SIR ROY CAMERON, Professor of Morbid Anatomy at University College, London, and chapters on precautions and first aid. The book concludes with a review of precautions against radiations from the Health and Safety Branch of the U.K.A.E.A., Risley, and a glossary of medical terms. Considerable efforts have evidently been made to achieve consistency with other workers in the field. For example, B.S. 2474:1954 has been followed for names of chemicals, and the Association of British Chemical Manufacturers' booklet on "Marking Containers" for the system of warning notices. In listing properties of toxic agents emphasis has been given to appearance, volatility, smell and — an ingenious, very practical point this—to the solubility, miscibility or reactivity with water because of the wide use of water in first-aid measures. The action to be taken is classified in a standard order, as applicable, under side-headings of "vapour inhaled", "affected eyes", "skin contact", "if swallowed". The book is strongly bound, and should stand up to frequent handling. It is a pity that the editor decided not to provide an index for the general review sections, but this very helpful volume can nevertheless be highly recommended for use in all places where harmful chemicals may be encountered.

I. BERKOVITCH

Low Temperature Techniques
by F. Din and A. H. Cockett
George Newnes Ltd., London, 40s.

A MORE representative title for this book would be "A plain man's guide to low temperatures". The word "techniques" normally connotes a detailed presentation of the actual constructional methods and experimental procedures employed in the chosen subject, garnished with an adequate quantity of know-how. Thus in discussing low-temperature techniques in the laboratory specific guidance would be expected on the construction of syphons, glass-to-metal seals and on low-temperature vacuum leaks. Similarly, in industrial low temperatures instruction would be expected on the jointing between metal components and cold box construction and insulation. None of these topics is dealt with.

In essence, the material presented is a review of the principles underlying methods for achieving and measuring low temperatures together with a largely qualitative description of the ways in which low temperatures are employed in the laboratory and in industry. Whilst both authors are employed in an industrial research and development laboratory, this fact does not bias the content of the book.

A brief but useful account of the history of low temperatures is given. It is followed by a survey of methods of producing low temperatures including descriptions of the Philips and Collins machines and an introduction to adiabatic demagnetisation. The section on the measurement of low temperatures is well written and includes constructional details of the thermometers together with calibration data. The chapter on properties of material at low temperatures bring together a great deal of useful information, but the absence of specific references for the data and the brevity of the discussion on mechanical strength (no mention of fatigue!) limit its value. The remainder of the book provides a short but well-informed description of current industrial plant for low-temperature gas separation and liquefaction.

The arrangement of material is good, the presentation is simple and could be followed by anyone qualified in science to "advanced-level" standard. Thus the book will be much appreciated by the interested layman and also by the practising engineer who finds himself involved with low temperatures.

G. G. HASELDEN

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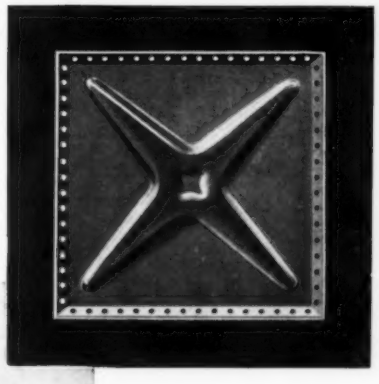
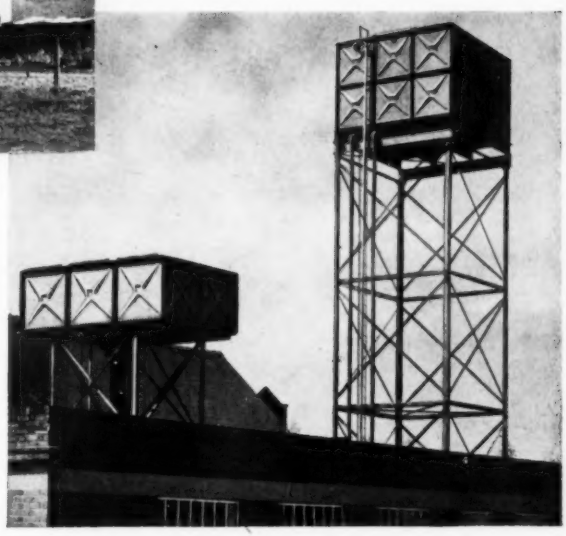


Pressed Steel Storage Tanks



imple to erect with all tools provided, these sectional tanks ensure reliable storage for water, heavy fuel oils and other liquids. Available in capacities from 220 gallons upwards. (above) A covered water tank for from 19,000 gallons, 20′ x 20′ x 8′ deep, supplied to a housing estate. (right) The two other tanks illustrated (6,700 gallons and 2,200 gallons) serve the boiler house at a modern hospital.

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Abstracts

The Transition from Free to Forced Convection in Mass Transfer from Solid Spheres*

Mass transfer rates have been measured at 30°C over a Reynolds number range of 1 to 130 for the solution of ¾-in.-diameter spheres of benzoic acid for both upflow and downflow streams. These are compared with similar results for ¼-in.-diameter spheres and show a gradual transition from forced to free convection. The interaction between the two effects is complex but less marked for the smaller spheres. Free convective effects do not disappear entirely until a Reynolds number of about 250 for the ¾-in. spheres, compared with a value of 750 given for ¼-in. spheres in previous work.

F. H. Garner and J. M. Hoffman, *A.I.Ch.E.J.*, 1960, **6** (4), 578.

Mass Transfer between Sprayed Film and Counterflowing Vapour in Two-substance Mixtures*

Mass exchange in rectification of ethanol/water mixtures has been measured under conditions which were accurately controlled from the kinematic and geometrical point of view. The experimental results show that the mass exchange primarily depends on the state of flow in the vapour. In the range investigated an exponential equation applies relating the Nusselt number of the second type, the Reynolds number for the vapour flow and the Schmidt number for the vapour.

H. P. Low-Fraulin, *Chem. Ing. Tech.*, 1961, **33**, 39.

Unsteady Multi-component Diffusional Evaporation*

The unsteady diffusion-controlled evaporation of a pure fluid into a multi-component mixture is studied by a boundary-layer method. An expression is obtained for the volume of vapour produced by the evaporating species as a function of time. It is shown that the result can be interpreted in terms of an effective diffusivity of the type proposed by Wilke and by Stewart. It is further demonstrated that the boundary-layer method predicts a sweep-diffusion effect.

Hsien-Wen Hsu and R. Byron Bird, *A.I.Ch.E.J.*, 1960, **6** (4), 551.

Reaction Rate and Diffusion

The author presents a chart for calculating the decomposition in reactions which are heterogeneous, unimolecular, constant volume and first order, as a function of the rates of reaction and diffusion.

J. Stricknodt, *Chem. Ing. Tech.*, 1961, **33**, 43.

GROUP D — PHYSICAL SEPARATIONS

The Role of Porosity in Filtration: IV. Contrast Pressure Filtration*

Certain assumptions which have previously served as a basis for the conventional equations employed in constant pressure filtration are shown to be in error. It is demonstrated that the specific filtration resistance, the ratio of the mass of wet to mass of dry cake, and the rate of flow, $q = dv/d\theta$, are not constant as has been assumed. In an example it is shown that q undergoes an eightfold variation as the liquid flows from the cake surface through to the medium.

Since the product αq appears in the basic differential equation, incorrect values of q lead to errors in the calculated values of α arising from experimental data. The errors are significant when thick slurries are employed.

New partial differential equations are presented for flow through compressible media in which q varies with cake thickness. Modifications of the conventional constant-pressure equations are presented.

F. M. Tiller and H. R. Cooper, *A.I.Ch.E.J.*, 1960, **6** (4), 595.

The Efficiency of Fibrous Aerosol Filters: Deposition by Diffusion of Particles of Finite Diameter*

A semi-theoretical expression for the filtration efficiency of fibrous aerosol filters operated at low velocities is tested with new data taken from the literature and is presented in a simplified form for design calculations. The existence of a minimum filtration efficiency is discussed and theory and data are compared.

R. E. Pasceri and S. K. Friedlander, *Can. J. Ch. E.*, 1960, **6**, 212.

* Author's abstract.

Filter Cake Washing Performance*

Differential equations for a filter cake wash cycle are derived on the assumption that the wash liquor executes plug flow in the pores of the cake, with continuous mass transfer between the liquor and a boundary film of filtrate. These equations are solved and the solutions presented as performance charts. The solutions are compared with certain experimental results.

M. T. Kuo, *A.I.Ch.E.J.*, 1960, **6** (4), 566.

Modern Concepts of Crystallisation

The author discusses the current theory and practice of the modern draught tube and baffle crystalliser. Crystals of commercially acceptable size can be produced by removal of excess fines, circulation of crystals to the supersaturation zone and maintenance of high magma density. An additional factor, retention time, is related to these three factors.

H. B. Caldwell, *Ind. Eng. Chem.*, 1961, **53** (2), 115.

GROUP E — PROCESSING

Formation and Quenching of Nitric Oxide in a Continuous Flow Furnace

The authors describe a pre-pilot plant unit for the thermal fixation of atmospheric nitrogen in a zirconia brick furnace; gas temperatures over 3000°K were reached, but the quenching rates were insufficient to fix the nitric oxide at such high temperatures. Even so, yields of 3.7 per cent nitric oxide, by volume, were obtained.

A separate water-cooled sampling probe, inserted at the top of the furnace, gave quenching rates of the order of 10^6°C/sec and the effect of variation in the quenching rate, while maintaining other conditions constant, was studied. Oxygen concentration appeared to have less influence than previous results had indicated, but this may be due to uncertainties in estimating gas temperatures near 3000°K.

D. L. Mordell, A. L. Thompson and G. Yano, *Can. J. Chem. Eng.*, 1960, **6**, 206.

Macroscopic Mixing in Fluidisation

An account of mixing studies carried out in beds of lead and glass particles fluidised with water.

The mixing data for the central portion of the bed yield radial eddy diffusivities and scales and intensities of turbulence. For the same void fraction both the eddy diffusivity and scale of turbulence are larger for lead than for glass particles, but the velocity fluctuations appear to be independent of the density of the particles. Minimum Peclet numbers are observed at a fraction void of about 0.7. The work shows that there are large-scale eddies in a fluidised bed whose size is much larger than a particle diameter. The interpretation of the mixing data is coupled with visual observations of fluidised-bed flow patterns.

E. J. Cairns and J. M. Prausnitz, *A.I.Ch.E.J.*, 1960, **6** (4), 554.

Problems in the Electrolytic Production of Metals from Fusions, illustrated by Fusion Electrolysis of Alumium

The relationships which exist in the electrolysis of fused salts are at present less obvious than those in the electrolysis of aqueous solutions. The paper discusses this problem in detail, using as example the fusion electrolysis of aluminium. Recent investigations have somewhat clarified the conditions existing in the fusions of the system NaF/AlF_3 and Na_3ALF_6/Al_2O_3. Ideas, based on experience obtained in practical operation, are presented on the reduction energy, ion mechanism, current yield, carbon consumption and heat and energy balances of the electrolytic cell system.

H. Ginsberg, *Chem. Ing. Tech.*, 1961, **33** (2), 80.

Problems in Electrolytic Production and Refining of Metals from Aqueous Solutions

The author describes advances in the field of electrolytic metal production and refining; namely, the deposition of metal powders or coherent layers having definite surface properties, the deposition of metal alloys and the production of high purity metals and semi-conductors by the amalgam process.

O. Emert, *Chemie. Ing. Tech.*, 1961, **33** (2), 93.

Recovery Ammonia from Ammonia-water Vapours

The paper presents several alternative methods for processing these vapours. The best method was found to depend on the vapour feed composition for each case and the most suitable methods, for feeds ranging in composition 0-100 per cent ammonia, are discussed.

M. C. Fields, *Ind. Eng. Chem.*, 1961, **53** (2), 131.

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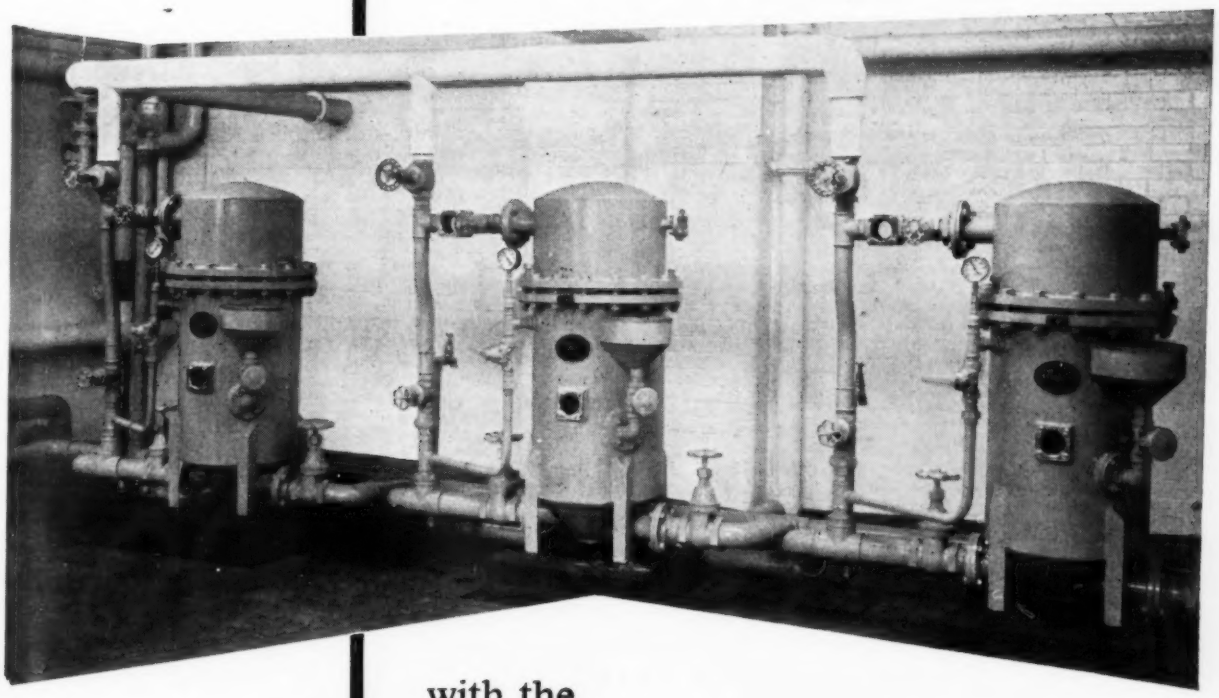

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Abstracts

Preparation of Carboxymethyl Celluloses of Low Substitution Degree

The author reviews the main lines along which carboxymethyl celluloses are produced industrially. He then specifies the various physicochemical transformations which take place in the reacting mass during the course of successive manufacturing operations. He also shows what influence the preparation process has on the properties of the products obtained, and concludes that the operations may be simplified considerably by the intense milling of the reacting mass during carboxymethylation.

M. Bouttemy, *Chimie et Ind.*, 1960, **84** (6), 891.

A Computational Model for Predicting and Correlating the Behaviour of Fixed-bed Reactors

A mathematical model, based on a two-dimensional network of perfectly stirred tanks, is developed for predicting the mixing characteristics of fixed beds of spheres. This model is also extended to cover chemical reaction in a cylindrical fixed bed of porous catalyst spheres. A simplified system is developed to indicate the practical advantages of the model.

I. "Derivation of Model for Non-reactive Systems." H. A. Deans and L. Lapidus, *A.I.Ch.E.J.*, 1960, **6** (4), 657.

II. "Extension to Chemically Reactive Systems." *A.I.Ch.E.J.*, 1960, **6** (4), 663.

Single Tower, Low-pressure Absorption Plant for Production of Nitric Acid from Nitrous Gases

An absorption tower for low-pressure conditions has been developed from theoretical considerations. In this tower, the tray spacing is greater in the upper section and perforated plates are incorporated in the space between the trays. Very favourable absorption conditions are obtained even with a small number of trays.

Pressure-gas Heated Waste-heat Boilers for Dirty Gases

The author describes the constructional features of a tower-tube boiler plant, operating at 15 atm on the gas side, for cooling dirty gases. There are three different processes for cleaning the tower-tubes.

F. Vollhardt, *Chem. Ing. Tech.*, 1961, **33**, 37.

Methods of Recovery of Acetic Acid

The author presents an historical outline of the problem and a detailed description of the various processes used: azeotropic distillation, extractive distillation, liquid-liquid extracton.

D. F. Othmer, *Chimie et Ind.*, 1960, **84** (6), 891. Published in German in *Dechema Monographien*.

GROUP F — PROCESS CONTROL

A Wet Test Meter for Low Flows and High Pressures

This article describes the development of a high-pressure wet test meter for measuring hydrogen at rates as low as 0.2 standard cu. ft per hour at pressures of 1000 psig. The meter operated satisfactorily for more than 2000 hours.

W. M. Miller, *Ind. Eng. Chem.*, 1961, **53** (2), 127.

GROUP G — MISCELLANEOUS

Pressure Drop and Power Requirements in a Stirred Fluidised Bed

Observations on the effect which stirring of fluidised solids has on the pressure drop are reported for a 6-in.-diameter, 12-in.-high unit fitted with a special adjustable blade stirrer.

Blade positioning relative to sense of rotation had a very profound effect on pressure drop and on power requirements. When the solids circulation pattern was similar to that found in a dense phase fluidised charge, the resulting pressure drop was always considerably lower than the conventional unstirred fluidised-bed pressure drop. The power requirements for this type of stirrer were lowest. For other stirrer design and other induced solids convection patterns, the pressure drop was less influenced and the power requirements were much greater.

Solids used were a relatively coarse silica sand for which both pressure drop as well as power requirement data were reported. Power requirement data were also obtained for a much finer alumina powder. These were lower than for sand.

M. Leva, *A.I.Ch.E.J.*, 1960, **6** (4), 688.

Similarity in Chemical Reactions

Two chemical processes are similar if they can both perform the same conversion taken as the reference. The reactors in which these processes are carried out may operate continuously or intermitently, in homogeneous or heterogeneous phase.

Starting from kinetic data, the author reviews the various methods for designing homogeneous reactors and shows how, in the absence of such data, the reactor may still be scaled-up. It is always possible to change from a discontinuous operation to a continuous one by using a tubular reactor in conditions of turbulent flow.

A continuous, stirred, tank reactor should not be used except in the case of simple reactions.

P. Trambouze, *Gen. Chim.*, S.1960-6-189.

The Corrosion Behaviour of Aluminium in Natural Waters

A review of the behaviour of aluminium in contact with natural waters discusses the influence of water composition and service factors on corrosivity to aluminium. Laboratory pitting test methods are described and data are presented to support the existence of a cube root (in time) rate curve for the pitting of aluminium in water. Information on the pitting of aluminium in 17 Canadian waters is given and mention is also made of the pitting of aluminium in seawater and oil field brines. A statistical treatment of pitting data is presented. Finally, measures for extending the life of aluminium equipment subject to pitting by water are suggested.

H. P. Godard, *Can. J. Chem. Eng.*, 1960, **5**, 167.

Time-saving Chart for Pipe Wall Thickness Selection

By means of a chart presented for the rapid determination of steel pipe wall thickness, given temperature, pressure and pipe size, corrosion can be taken into account and the method of constructing similar charts for other pipe materials is outlined.

W. P. Hansen, *Petrol. Engr.*, 1960, **32** (6), C14.

How to Design Pipe Supports for Petrochemical Plants

Aspects of support design are considered, such as their spacing, materials and construction. A number of design criteria are given, including weights, friction, expansion, wind and earthquake forces, ice loads, foundations, etc.

B. Laan, *Petrol. Engr.*, 1960, **32** (6), C50.

Corroded Tank Bottoms Repaired with Plastic

Glass fibre matt impregnated with liquid polyester resin is an effective means of repairing corroded storage tanks and similar installations. How to obtain satisfactory results, with a large saving in cost, is outlined in this paper.

D. Gavitt, *Petrol. Engr.*, 1960, **32** (7), D12.

Lobed Pressure Vessels

An unconventional form of pressure vessel is described in which pressure is contained within a spacial structure whose members are bridged by membranes of appropriate curvature called "lobes". The developments which lead to the proposal and study of this form of vessel are outlined and some associated theoretical and practical facts are considered. The design, construction and testing of two steel laboratory models of lobed vessels are reported and the significant conclusions reached in these studies are summarised.

J. P. Duncan and N. W. Murray. Paper 6 presented to a Symposium on Pressure Vessel Research towards Better Design, arranged by The Institution of Mechanical Engineers in co-operation with The British Welding Research Association.

The Use of Tritium to Study the Solubility of Water in Hydrocarbon Liquids

A study of water-hydrocarbon systems in equilibrium with an air stream, at different levels of humidity, below and up to saturation, using tritiated water as a tracer and the liquid scintillating technique of counting.

Results for benzene and toluene show that Henry's law is obeyed up to saturation. Total solubilities obtained at 20°C are 52 and 46 mg/100 g respectively.

Vapour radioactivity is shown to be 13 ±2 per cent lower than that of the solution in the temperature range 5-20°C.

B. D. Caddock and P. L. Davies, *J. Inst. Pet.*, Dec. 1960, p. 391.

Constructional Features and Possibilities for the Application of Chemical Pumps

A survey of special designs and constructional features of some chemical pumps is presented. Pumps with sliding ring seals, pumps with split tube motor drives and process pumps are described. Practical experience in application is reported and the possibility of interchanging individual parts is discussed.

F. H. Middeldorf, *Chem. Ing. Tech.*, 1961, **33**, 31.

Brit. Chem. Eng. Abstracts: **FOLIO No. 4**

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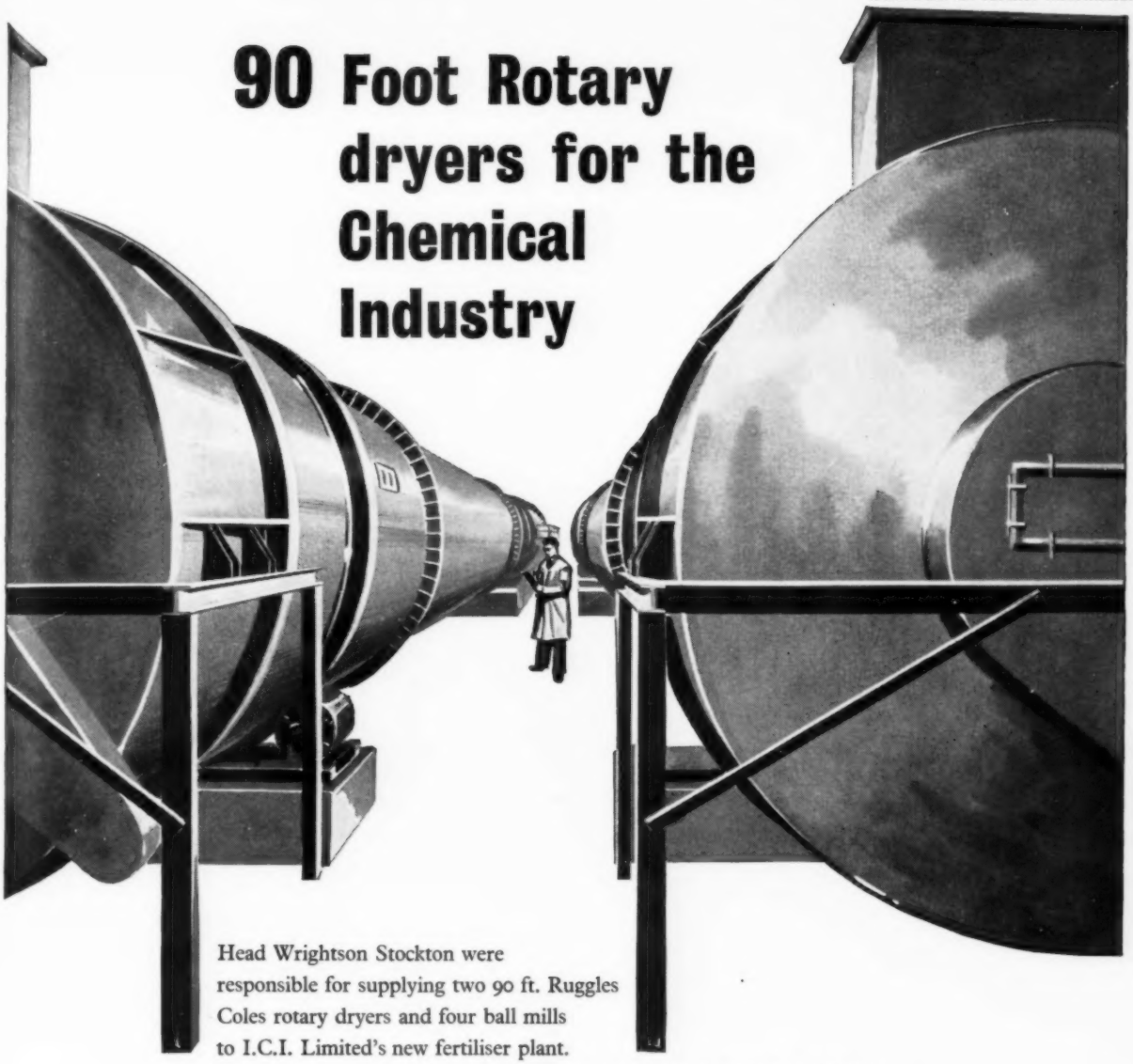

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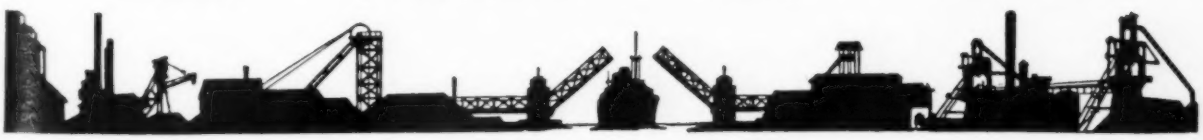

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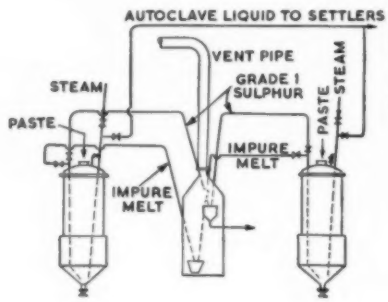
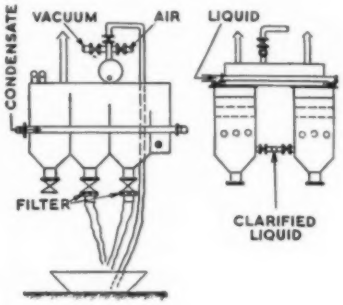
PROCESS ENGINEER'S NOTEBOOK

Improvements to Sulphur Recovery Plant





THE FIRST FEW months of operation of a sulphur recovery unit at a coke oven plant revealed a number of defects in the design of the unit. The sulphur is removed from the gas by the arsenic-soda process and the part of the plant which required the most alteration was the autoclave section. The autoclaves had been installed without settling tanks, bottom outlet connections were provided for discharging the impure melt and the resultant liquid product; the latter was then passed to filter presses.

Among the disadvantages of this layout were: damage to structural steelwork and other metallic objects exposed to the action of the gases released when the autoclave valves were opened; danger to operators through having to unblock the discharge valves which clogged frequently; while the lifting of moulds of impure melt discharged from the autoclaves, which the process made necessary, was a labour-consuming operation.

The following successful steps were taken to improve this situation. The first line of attack was to discharge the impure melt from the autoclaves by taking advantage of the operating pressure (2-2.5 atm). The impure melt feeds into a tundish fitted inside an exhausted hood, in which is also fitted another tundish for collecting high-quality sulphur. This sulphur is fed from this point to metal channels which allow the sulphur to be fed direct to tank cars. The autoclave liquid was fed by the pressure in the autoclaves to the first section of a tricompartment settling tank, of which there are two. The liquor passes from one end to the other, becomes clarified and before filtering enters another additional piece of equipment—a foam collector.

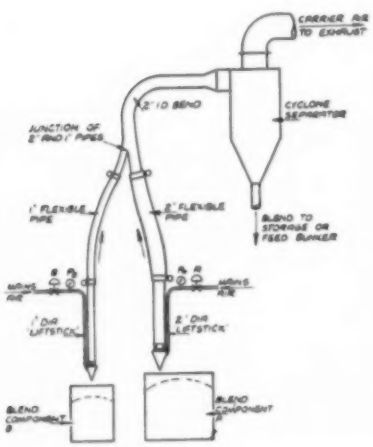
When, after a few weeks of operation, a settler becomes filled with debris it is taken out of operation. The residual liquid is then drained into a trough fitted beneath the settler discharge valves, and is drawn into an evacuated vessel located above the settlers; the vacuum in this vessel is broken to release its contents to the other settler now in operation. To prevent the valves in the transfer system from choking, grid sections are fitted downstream of the discharge valves at the settler run-off connections. The rubbish remaining in the out-of-operation settler is removed through further connections to dumpers.

(A. I. VOSKOBOINIK, *Coke and Chemistry*, USSR, 1960, 9, 32.)

An Experimental Pneumatic Blender for Granular Solids

AN EARLIER NOTE, entitled "A Portable Pneumatic Elevator for Granular Solids",[1] described a device (called a "lift stick") which had been used for refilling a solids feed bunker of a pilot plant directly from supply containers. Since then further development has shown that this system could be used as a convenient method for disperse phase blending of two granular solids on a pilot-plant scale.

[1] *Brit. Chem. Eng.*, June 1960, Process Engineer's Notebook, 441.



It had been observed that the rate of solids delivery from supply containers was a function of the nozzle air pressure; i.e., for given dimensions of "lift stick" constant rates of solid delivery could be obtained at constant pressure settings. The work being carried out demanded that two 10 B.S.S. coals (A and B) be blended continuously (with proportions of coal B in the range 5-20 per cent) to the feed bunker of the pilot plant. The diagram shows the arrangement of the system adopted for the purpose. For the major blend component, A, a 2 in. dia. "lift stick" was chosen; for minor component, B, a 1 in. dia. lift stick was made. The two lift sticks were connected to a common cyclone separator by means of flexible hoses and a junction piece which was designed to give smooth combination of the two streams. At the cyclone the blended solids were reclaimed from the carrier air stream and passed to the feed bunker of the pilot plant. Air was supplied to the jets of each "lift stick" at pressures which could be controlled independently by valves A and B and measured by gauges P_A and P_B. Some pre-calibration of the system was necessary to determine the pressure ratios P_A/P_B to give the required order of solid ratios A/B. For operation it was then only necessary to set the pressures P_A and P_B at the predetermined value to give the required solids ratio. Some results are given below:

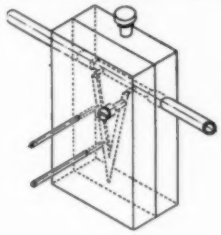
Lift Stick Pressures lb/sq in.		*Solids Ratio*
A	B	*A/B*
35	30	5.4
35	30	5.5
35	30	5.4
35	18	13.7
35	18	13.9
35	18	13.5

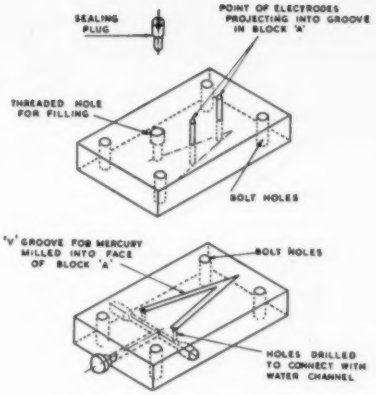
W. H. NEWTON and G. O. DAVIES

Simple Cooling Water Switch

THIS SIMPLE SWITCH is readily made from two slabs of perspex and its function is to act as a safety device for equipment requiring cooling water. Thus, if used in conjunction with a diffusion pump, circulating water failure would cause the pump to be switched off, or in the case of cooling water to bearings of high-temperature pumps an alarm would be given.

One face of the slab is drilled and tapped for the water channel, this being made close to one surface of one of the perspex blocks. A deep "V" groove is then cut in the surface in such a way that it cuts two holes drilled to connect with the water channel. A tapping is also made into the water channel at a point midway between the "V" connection for a bolt which serves as a throttle. In the remaining slab two platinum electrodes are




SEALING PLUG
POINT OF ELECTRODES PROJECTING INTO GROOVE IN BLOCK 'A'
THREADED HOLE FOR FILLING
BOLT HOLES
'V' GROOVE FOR MERCURY MILLED INTO FACE OF BLOCK 'A'
BOLT HOLES
HOLES DRILLED TO CONNECT WITH WATER CHANNEL

cemented and are arranged so that they fit into the upstream limb of the "V". The two blocks are bolted together to form a sealed unit.

In operation the cooling water passes through the channel and the screw adjusted to produce a pressure drop indication in the "V". The mercury makes contact with the electrodes when the required flow rate is achieved. On failure, the mercury level falls, the circuit is broken, and the equipment being cooled switched off.

Porous Stainless Panels Increase Bin Flow

VIBRATORS OR SHAKERS which normally would have been satisfactory in providing a steady flow of material proved totally ineffective in inducing the flow of solids from bins at a synthetic fibre plant. The material persisted in bridging and flow was persistently unreliable. This was serious, apart from its effect upon production, since a belt weigh feeder system was employed. A new approach was needed.

An attractive solution to the problem was aerating or fluidising the powder by means of inert gas or compressed air blown into the packed, almost-solid powder. In this particular instance the inert gas had to be nitrogen. This was to be done by blowing the nitrogen gas through porous stainless-steel panels lining the bin to provide a gaseous coated frictionless bin wall and by blowing additional nitrogen into the packed powder by means of perforated probes.

Initial trials were made with a variety of materials, including porous stone and woven fabric, but these proved unsatisfactory. Non-uniformity and contamination of the product due to impurities and linting prevented the use of these materials.

Investigation showed that large panels of uniform porosity sintered porous stainless steel possessed the right combination of properties needed for creating and dispersing a multitude of fine gas bubbles. The properties of PSS offered a prospect of zero product contamination while the uniform porosity with controlled pressure drop offered economy in nitrogen consumption. Only a quarter as much nitrogen was found to be required with the stainless panels compared with that required by other porous materials.

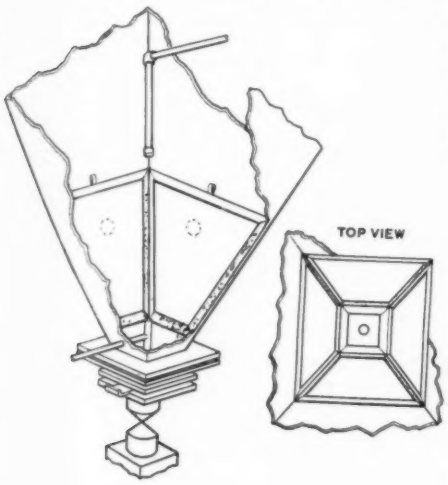
The design called for the use of four porous stainless-steel panels in the throat of each bin, and in addition, for porous probes to be placed farther up the bin wall and along the valley angles to prevent possible localised bridging. The porous panels were to be installed without removing any sections of existing bin walls and were, therefore, "hung" on the existing walls. The only cut made in the existing walls was for the gas supply. (See diagram.)

Once the design was settled, and the fluidising panels installed, the bins were filled with product. Gas was then fed to the panels and probes, and the discharge valve was opened. The flow of solids was instantaneous, the powder was fluid, and the flow to the weigh feeder was smooth and even.

Final gas rates established after a series of runs made to find the optimum gas quantities for the probes and panels were as follows:

Probes	...	0.2 cfm each
Panels ...	...	0.5 cfm each

A run was made to determine the ultimate capacity of the bins with the new system. However, due to instrumentation limitations, the bin discharge valve could not be opened beyond the two-thirds "open" position, limiting the flow rate available. Nevertheless, it was apparent that by opening the valve completely a higher flow could easily be attained, much greater in fact than the threefold increase which was actually achieved.



By courtesy of the Pall Corporation, Glen Cove, New York, U.S.A.

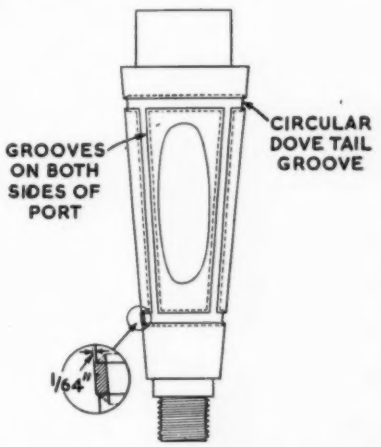
Improved Operation of Metal Stopcock

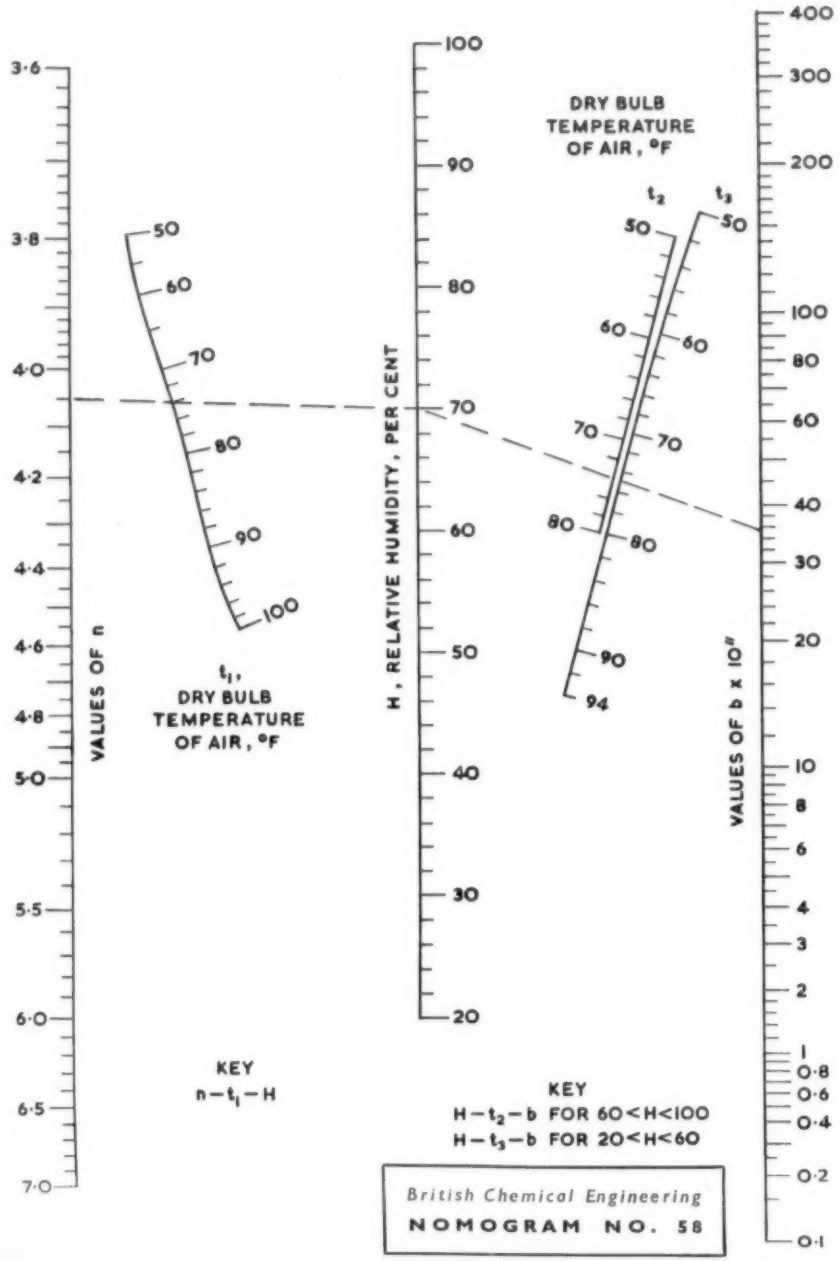
STICKING PLUGS OF STOPCOCKS in pipelines carrying processing fluids is a not uncommon occurrence. One source of the trouble is the need to have a perfect fit between plug and valve body to prevent leakage. If the fluids tend to corrode the valve material the faces often seize.

A remedy is to fit around the port openings in the plug strips of P.T.F.E. Grooves are machined into the plug circumferentially above and below the port and longitudinally along the taper to form a junction with the circumferential grooves. The grooves must be dovetailed to retain the strips which themselves project $\frac{1}{64}$in. beyond the periphery of the plug.

Before the plug is replaced in the body, any burrs or grooves in the taper surface must be removed, otherwise the plastic inserts will be damaged.

(Based upon a note appearing in *Metalworking Production*, 1961, 105, 5, 71.)





EVAPORATION FROM PONDS AND THICKENERS

by D. S. DAVIS

(Head, Department of Paper and Pulp Technology, University of Alabama)

The rate of evaporation of water from heated ponds and thickeners, common in many metallurgical and mineral-dressing processes, can be calculated by means of the equation[1]

$$w = 2.387\ (1 + 0.330V)\ b\ t^{n} \qquad \ldots . (1)$$

where w = rate of evaporation of water, lb/(hr) (sq ft);
V = wind velocity, mi/hr;
t = temperature of the water, °F;
b is defined implicitly by the equation

$$\log\ (b + 0.036 - 0.0021\ b^{-0.339}) = f + g\ H \qquad \ldots . (2)$$

$$n = 3.000 - 10^{r + s\ H}; \qquad \ldots . (3)$$

H = relative humidity of the atmosphere, percentage;

and f, g, r and s depend upon the dry bulb temperature of the atmosphere.

The values of b and n can be determined with ease from the line co-ordinate chart of Fig. 1, which is based on Equations (2) and (3), developed specifically for the design of this chart. Fig. 1 was constructed in accordance with methods described previously.[2] The rate of evaporation can be read quickly and accurately from Fig. 2, which is based on Equation (1) and was designed through application of standard methods.[3]

Use of the charts is illustrated as follows: What is the rate of evaporation of water from a pond maintained at 110°F when the dry bulb temperature of the surrounding air is 74°F, the relative humidity is 70 per cent and the air is moving at the rate of 5 mi/hr? Follow the key at the left in Fig. 1; connect 70 on the H-scale and 74 on the t_1-scale with a straight line to intersect the n-scale at 4.05. Then follow the key at the right and connect 70 on the H-scale and 74 on the t_2-scale with a straight line to intersect the b-scale at 35×10^{-11}. Then follow the key in Fig. 2 and connect 110 on the t-scale and 4.05 on the n-scale with a straight line to intersect the α-axis. Connect this intersection and 5 on the V-scale with a straight line to intersect the β-axis. Connect this intersection with 35×10^{-11} on the b-scale with a straight line to intersect the w-scale at 0.40 lb/(hr) (sq ft). If the value of b had been one-tenth that of this illustration, the value of w would have been 0.040 lb/(hr) (sq ft).

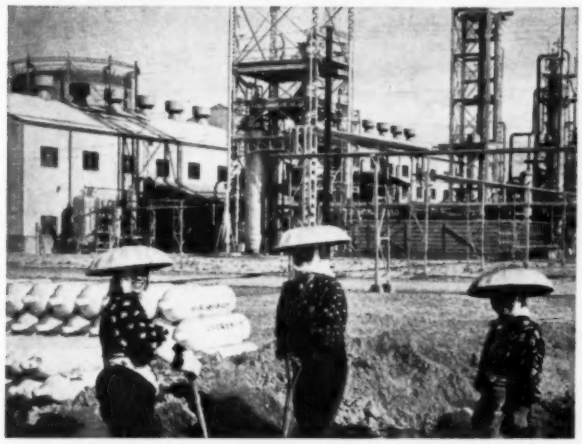
REFERENCES

[1] Thomas, B. L. *Chem. Eng.*, **67** (16), 129 (1960).
[2] Davis, D. S. "Nomography and Empirical Equations", Chap. 10, Reinhold Publishing Corporation, New York, 1955.
[3] Davis, D. S. "Nomography and Empirical Equations", Chap. 9, Reinhold Publishing Corporation, New York, 1955.

BCE 9425 for further information

AMMONIA

NEW CHEMICO PLANTS TO BOOST WORLD CAPACITY 12½%



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- 8 NEW PLANTS IN THE U.S.A.
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Since the first months of 1957, 13 new Chemico ammonia plants have been completed and put into operation. Another 7 Chemico plants are under construction or in the engineering stages. The design capacity of these 20 plants totals more than 1,000,000 tons per year.

Chemico's versatility in ammonia plant design is evidenced by the variety of raw materials on which these plants are based. While natural gas was the most common, crude oil, fuel oil and coke oven gas were also used. Regardless of feed stock, however, all thirteen of the operating plants were completed on schedule and started up without significant difficulties or delays. One plant, designed to produce 150 tons per day, took less than ten months to build, from initial go-ahead to completion of a satisfactory test-run.

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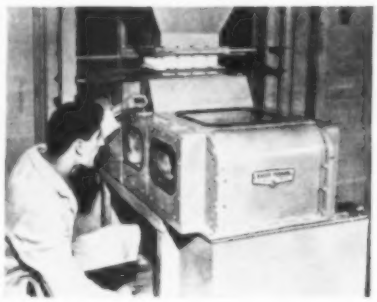
PLANT EQUIPMENT NEWS

Accurate Weigh Feeder



A new design of weigh feeder incorporates the solution to a number of problems. To achieve accuracy it has a short time cycle control loop. It is enclosed to prevent errors through collection of dust and draughts, and change in tension of the conveyor belt does not register as a weight change.

The machine also has a wide range of output and is sufficiently robust to withstand normal factory conditions without detriment to sensitivity.

These requirements are met by the new Baker Perkins weigh feeder whose main components are a hopper with a gate-type discharge of material to a fixed conveyor, beneath which is a weight sensing mechanism.

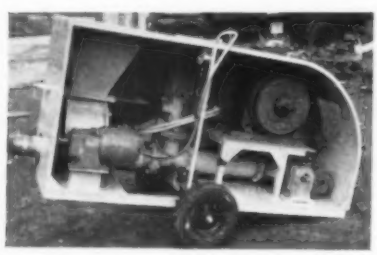
Accuracy is ±1 per cent, based on 1-min readings over any part of the range.

The machine can be fitted to deliver any output from 50 lb/hr to 5 tons/hr, maintaining the accuracy stated, and any individual machine can have approximately 10:1 output range within those limits. **Baker Perkins Ltd., Westwood Works, Peterborough, Northants.**

BCE 9460 for further information

Mobile Pneumatic Conveying Units

A capacity of up to 20 tons per hour is offered by a mobile conveying unit. Such materials as coal, limestone, cement, coffee and so forth are readily transferred by the unit. Pipe size required is 4 in. BSP and the Roots type compressor, which supplies fluidising air, delivers up to 350 cfm. The prime mover may be a 25-hp electric motor or a petrol or diesel engine.



The unit is provided with relief and non-return valves, and a rotary valve of the "Callow" Blower type for feeding solids into the transfer system. **F. W. Callow (Engineers) Ltd., Kirkby Industrial Estate, Liverpool.**

BCE 9461 for further information

Flow Indicator

This new vane-type flow indicator is suitable for use on pressure lines where flow is either horizontal or vertical. Illustrated is the Model 112 flow indicator showing approximate proportion of full flow. A fractional scale can be incorporated if desired. The six sizes obtainable fit pipes from ¾ in. to 2½ in. diam., are constructed of bronze and pressure tested to 100 psi.

Suba Hydraulics Ltd., Limes Place, Limes Road, West Croydon, Surrey.

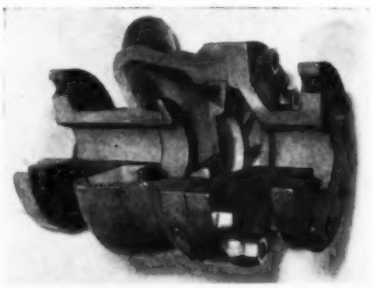
BCE 9462 for further information

One-moving-part Steam Trap

A trap, designed to discharge condensate at steam pressure consistently from no load to maximum pressure, has only one moving part and all internal trim is renewable in position. The body materials may be either manganese bronze forgings or stainless-steel castings, and the internal trim is of high-grade stainless steel. No gaskets are used and all mating surfaces are metal-to-metal contact. The manganese bronze trap is suitable for pressures ranging from 5-250 psig, and the stainless-steel version is suitable for pressures up to 400 psig. The traps are available with ½-in. or ¾-in. connections screwed with BSP or ANP threads. **Midland Industries Ltd., Heath Town Works, Wolverhampton.**

BCE 9463 for further information

Vortex Pipeline Separator



These separators extract moisture from steam and moisture and oil droplets from compressed air; they can also be used for separating out dirt particles from gases. They operate on the vortex principle, the vortex being created by impingement of the gas, steam or air on a vaneplate. The vanes are specially designed to suit each individual application, so that there are no moving parts and therefore no maintenance requirements.

Standard models are available for vertical downward and horizontal installations; it is also available for upward flow with a small modification and can therefore be used for exhaust heads. Pressures up to 150 psi are handled with cast iron, to 500 psi with cast steel, to 1500 psi with cast alloy steel and above that figure with special steels. For corrosive duties and other special applications materials can be specified by the customer. There is no reversal of flow and pressure drop can be maintained below 1 per cent. **J. H. Carruthers & Co. Ltd., East Kilbride, Glasgow.**

BCE 9464 for further information

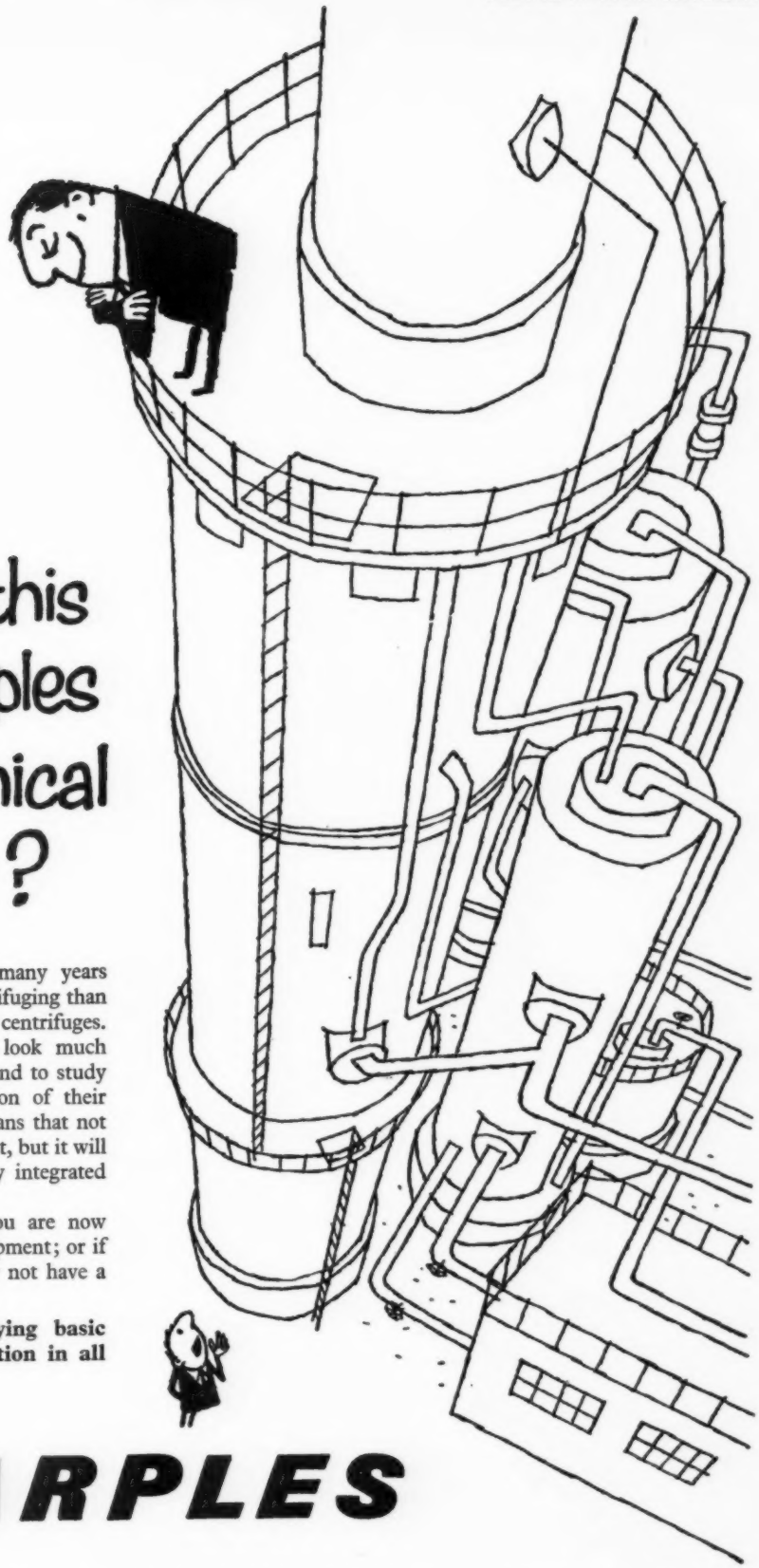
Special Slide Rule Calculators

The latest purpose-designed slide rule type calculator available is the "Pipe Weights and Strengths" slide rule. It will be very useful for constantly recurring routine calculations and gives the answers in seconds—in British and metric units.

One operation gives the weight of steel, aluminium, cast or spun iron, bronze, copper or lead pipes when the diameter and thickness are known. A second operation enables the user to determine the value of either the bursting, test or working pressure; the yield or stress point of material required; or the O.D. or thickness of any metal pipe when the other three values are known. Finally, a set of scales gives the relative carrying capacities of pipes of different internal diameters. It is also possible to use the rule for straightforward price calculations. **Blundell Rules Ltd., Regulus Works, Lynch Lane, Weymouth, Dorset.**

BCE 9465 for further information

BCE 9427 for further information



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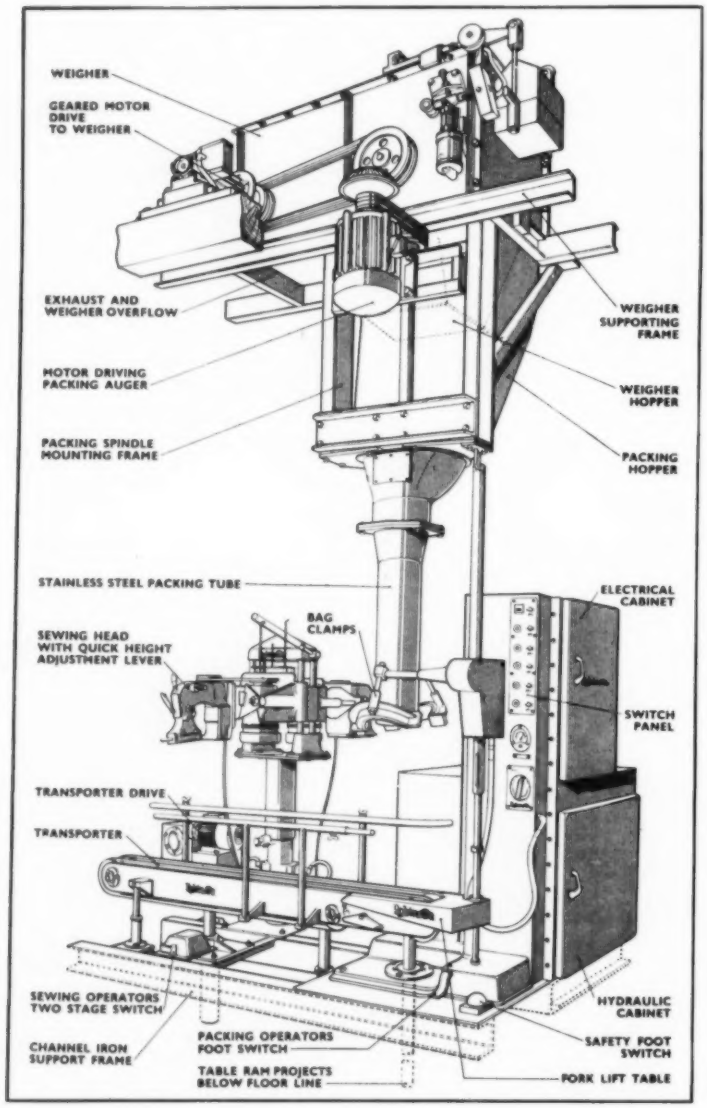
PLANT EQUIPMENT NEWS

Bag-filling Machine



An electro hydraulically-operated bag packer of up-to-date design achieves packing speeds on flour of eight bags per minute with 70-lb bags and six bags per minute with 140-lb bags.

In operation, when the geared motor incorporated in the weigher is energised the material to be weighed discharges into a pan until 80 per cent of the required load is delivered. A mechanism then comes into play which energises two thrusters, thereby closing a valve and reducing the solid feed to a trickle until the complete weight is delivered.

The machine, which is suitable for various types of bag containing 50 to 150 lb, has a number of safety features. For example, the fork-lift table, which supports the bag to be filled, is provided with a red foot switch so that the operator can control the movement of the table at any time during its upstroke. Simultaneously the bag clamps open immediately (see diagram). In addition, electrical interlocks are incorporated to prevent operation if a number of undesirable conditions occur. For example, the weigher may not be full or the main motor may stop.

Sewing equipment, with transporter from the weigher section, and a device to ensure uniform packing are among other noteworthy features of this machine. **Thomas Robinson & Son Ltd., Rochdale, Lancs.**

BCE 9466 for further information

Lead-clad Copper Tubing

The main field for this type of tubing is for applications like the heating or cooling of sulphuric acid and its solutions, phosphoric acid, chromic acid, sulphites and sulphates.

The process by which the cladding is done also enables copper tubing to be lead-lined. The coils treated in this way may be used at temperatures up to 260°C and at pressures up to 150 psig. Diameters of tubing which can be treated range from $\frac{1}{4}$ up to 2 in. Larger diameters can be treated, but this is a non-standard procedure; thickness of cladding provided is from $\frac{3}{32}$ in. to $\frac{1}{2}$ in. Here again heavier thicknesses can be provided as non-standard. Coils are provided in lengths of from 40-100 ft, depending on diameter. Straight lengths are normally 20 ft.

A simple technique has been worked out for joining lengths of tubing together. **David Brown Industries Ltd., Jackson Division, Manchester.**

BCE 9467 for further information

Continuous Registering of Gas Density

An instrument for continuously registering gas density, equipped with counterbalancing glass spheres, includes automatic compensation of pressure and temperature, whereby the density, as referred to the normal state, may be read without conversion. Under normal conditions, pressure correction is effected within a range of ± 20 in. water column, and temperature-correction within $\pm 27°F$. The measured values may be directly read in pounds per standardised cubic foot, while records are printed at 30-second intervals. Gas consumption is some 0.07 cu ft/min, and accuracy of indication ± 1 per cent of terminal value of the scale, within any measuring range, while suppressed zero point is also used. **Pollux GmbH, Ludwigshafen/Rh.**

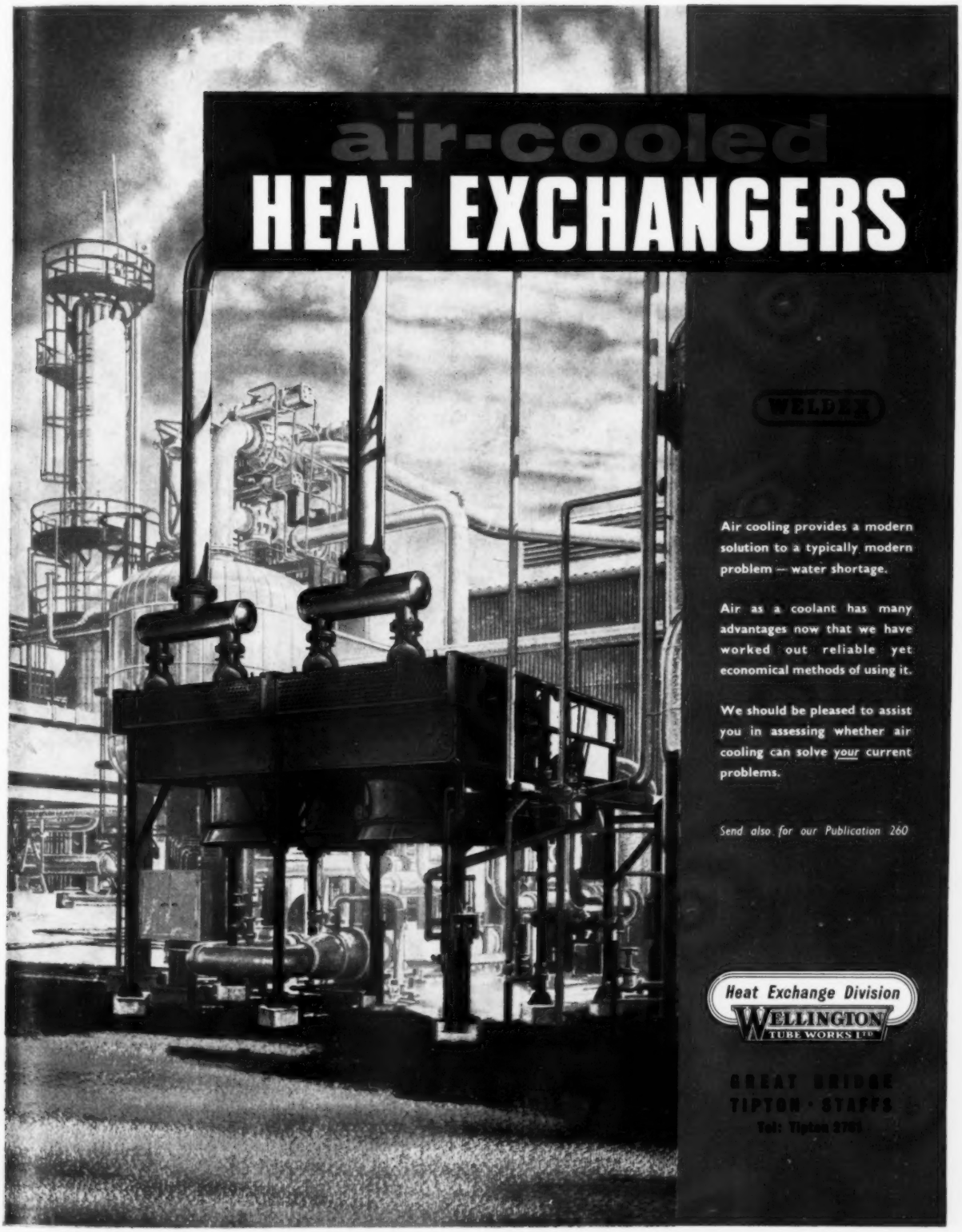
BCE 9468 for further information

Optimising Controller

Designed specifically for industrial process control applications, the Optimat is a small-scale single-purpose transistorised digital computer which accepts input signals from the process, and operates on the set points of up to three conventional three-term controllers.

The machine makes a small change to the set point of one of the variables and observes the effect of this change on both the specification and the criterion to be optimised. It then acts to establish the optimum operating conditions while maintaining the products within specification. As the optimum operating point changes due to uncontrolled variables such as demand, ageing of catalysts, quality of raw materials or ambient temperature, the computer continuously adjusts the process to the new optimum operating point. It may be connected directly to exising controllers on a plant. **Panellit Ltd., Borehamwood, Herts.**

BCE 9469 for further information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PLANT EQUIPMENT NEWS

Compact Filter

Although designed primarily as a filter for small quantity filtration in conjunction with machine tools, an addition to an existing range of streamline filters is also suitable for pilot-plant work.

Capable of superfine filtration (particle size selections of 3 microns and 12-14 microns respectively are typical), the filter elements work on the depth-type principle, whereby the fluid passes from the outside of a cylindrical porous element to the inside. In many elements the porosity decreases towards the interior, thus providing a stepped filtration and a high utilisation of the volume of filter material.

Element replacement can be rapidly performed and is a comparatively clean operation. This is made possible by a snorer attachment. Capacity is 2 gpm and maximum working pressure 50 psig. The filters can be lined with rubber or glass or, if necessary, made completely of stainless steel; a wide range of filter medium cartridges is available. **Par-Tool Engineering Co. Ltd., Filtration Division, Chobham Street, Langley Street, Luton, Beds.**

BCE 9470 for further information

Epoxy Flooring Composition

Heavy traffic and spilled corrosive liquids are sources of serious damage to factory flooring and lead to expensive maintenance. A new flooring composition consisting of an aggregate and special resins has strongly adherent properties and gives a coloured, non-slip surface resistant to many chemicals. Its abrasion resistance is exceptional, so it withstands heavy traffic conditions. **Tretol Ltd., Tretol House, The Hyde, London, N.W.9.**

BCE 9471 for further information

Powders for Refractory Spraying

Reliable commercial plasma spray units now available have extended the range of high-temperature materials which can be applied as coatings. Plasma equipment, producing flame temperatures up to 15,000°F with inert or non-reactive atmospheres, when necessary, has removed the limitations imposed by chemical flame sources so that, at least in theory, any material which does not decompose below its melting point can be sprayed.

Special powders for use with such equipment include carbides, borides and nitrides, in addition to the conventional oxides and metals.

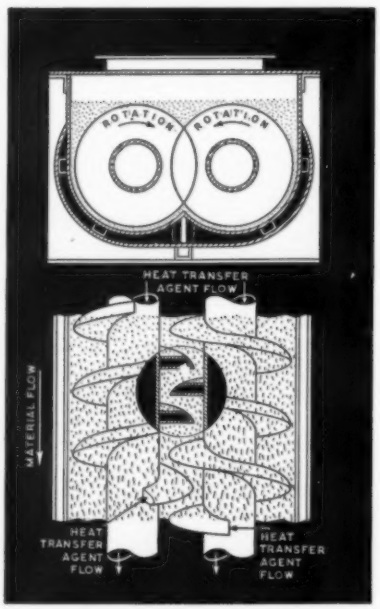
Powders of narrow particle size range (−150 mesh to +325 mesh) are now available, the materials being chromium boride, titanium boride, zirconium boride, norbide boron carbide (tech. grade), boron carbide, chromium carbide, Cr_3C_2, titanium carbide, zirconium carbide and titanium nitride. **Norton Grinding Wheel Co., Ltd., Welwyn Garden City, Herts.**

BCE 9472 for further information

Conveyor Heat Exchanger

The main feature of this design, developed originally in the United States, is a hollow screw through which the heat-transfer agent is circulated. At the exterior surface of the screw the material surrounding it is subjected to an interfolding and dispersing action which, in addition to giving high rate of heat transfer, keeps dusting and abrasion to a minimum.

Machines making use of this principle may have either double or quadruple screws. Diameters of screws are 7, 12 and 16, and lengths of trough in which they operate vary from 8 to 20 ft. Some idea of their capacity can be gleaned from the following figures. Thus for heating there have been applications where ammonium nitrate has been heated from 68-104°F for a throughput of 8000 lb/h. Cooling performance is shown also for ammonium nitrate, which can be cooled from 284-86°F at the rate of 10,000 lb/h.

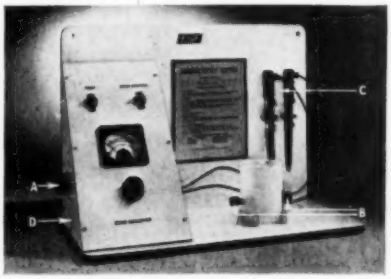


Apart from heating and cooling, drying is another important field for this machine, which is known as the "Holo Flite" processor.

Normally the trough working pressure is atmospheric, but troughs for higher pressures can be supplied. Where necessary, the trough may also be jacketed. **Holdan Engineers (London) Ltd., 119 Victoria Street, London, S.W.1.**

BCE 9473 for further information

Quality Control of Liquids



The correct assessment of boiler feed water, and generally speaking quality control of any aqueous effluent, is greatly facilitated by conductivity measurements.

The Elga portable conductivity meter (illustrated) is a self-contained unit with a range of 100,000 to 4 million ohm-cm³. Two cells are provided: a portable conductivity beaker for batch tests and a flow line cell for continuous measurements. The moving coil meter is energised by a 90 V dry battery. **Elga Products Ltd., Lane End, Bucks.**

BCE 9474 for further information

Glass Linings for Bunkers

Glass linings have been applied to coal bunkers with a consequent extension in the life of the bunker sides and reduction of maintenance costs. Operating advantages are provided by the reduced coefficient of friction which reduces the hang-up of fine coal on the sides of bunkers and provides a more even flow of material. In some instances the same capacity has been achieved with a lower headroom. **Dunlop Rubber Co. Ltd., Chemical Plant Lining Group, Cambridge Street, Manchester.**

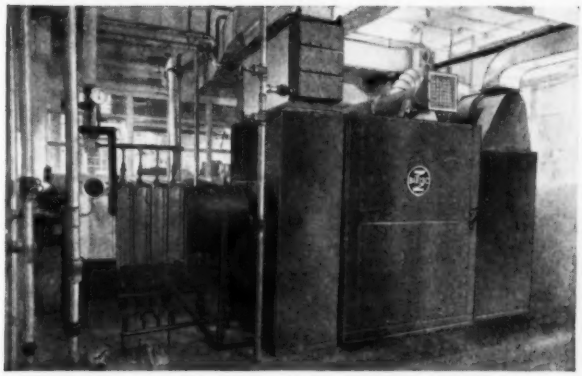
BCE 9475 for further information

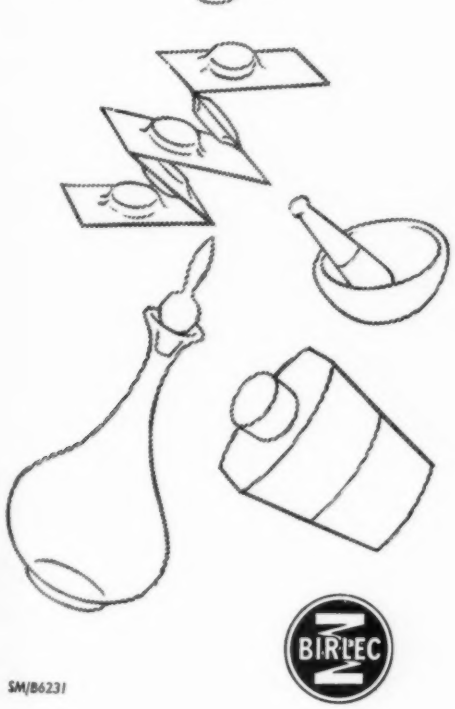
Console for Automated Process

Built with the guidance of Peabody Ltd., a recently-assembled graphic control console gives a step-by-step picture of all the stages of the final processing of titanium dioxide required for paint manufacture. The feed lines are represented by broad embossed channels and the flow lines by narrower channels. Each control point in the process is indicated so that any irregularities immediately become apparent. The control panel automatically

DRY HYGROSCOPICS

Model CO Dehumidifier installation at Reckitt & Sons Limited, Hull, for their Steradent process room.





Many chemicals and pharmaceutical products are harmed by atmospheric moisture. Damp weather may spoil both the product and the smooth flow of the production processes if atmospheric humidity throughout the plant is not controlled. Birlec Direct Dehumidifiers offer a simple and highly efficient means of holding the relative humidity of a process room or storage enclosure at a safe low level.

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PLANT EQUIPMENT NEWS

brings into operation safety devices and regulating mechanisms should any faults develop.

Similar panels fitted with as many as 52 solenoid-operated switches, each controlling a separate processing stage, have been supplied for other processes such as the manufacture of fertilisers, detergents and petroleum products. They are economical in space, since normal dimensions are 10 ft length, 6 ft 6 in. height and 3 ft 9 in. depth. **Lintott Engineering Ltd., Horsham.**

BCE 9476 for further information

All-plastic Non-return Valve

This "flap-type" all-plastic non-return valve is the latest addition to the Barflo range. The "flapper" is made of neoprene rubber and the body can be constructed from most rigid plastics. Most plastic tube sizes for vertical and horizontal operating can be fabricated, and most operating conditions met. **Barflo Ltd., 56 Cavendish Place, Eastbourne.**

BCE 9477 for further information

Nylon Bushings

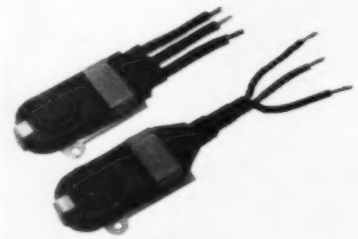
One hundred and sixteen standard sizes of nylon tubing ranging from 2 in. O.D. × 1 in. I.D. to 15 in. O.D. × 14¼ in. I.D. can be supplied from stock or on very short delivery for users requiring nylon bearings. The tubing has been specially developed for such applications and other similar applications such as guides, bushings and valve seats.

It is worth noting that lubricated nylon bearings will often out-perform orthodox phosphor-bronze bearings, and they have an advantage of being only one-seventh the weight of the equivalent-sized bronze bearing. **Polypenco Ltd., 68/70 Tewin Road, Welwyn Garden City, Herts.**

BCE 9478 for further information

Neoprene Circuit Breakers

The introduction of neoprene synthetic rubber as an insulant and protective



covering for these French-designed industrial circuit breakers enables them to function in dusty atmospheres, oils, water and chemicals. As illustrated, the "Minirupteur" circuit breaker is completely protected by neoprene, which is a good electrical insulator, and which can withstand ageing and weather far better than the plastic materials it replaces. **Société Sermec, 18 rue Jean-Jacques Rousseau, Valence, France.**

BCE 9479 for further information

Lightweight Crushable Ducting

A unique type of ultra-lightweight crushable ducting can be passed through existing pipework or metal ducting to render it corrosion resisting. This new ducting is able to reform to contour after crushing, and it can be made with built-in nylon corrugations to allow for misalignment. It is constructed from a specially developed resin compound reinforced with glass cloth. The brittleness previously associated with some glass-reinforced plastic materials has been overcome.

The cloth for this ducting is supplied by Fothergill & Harvey (Tyglas cloth) and the ducting is supplied by **Baxter, Woodhouse & Taylor Ltd., Poynton, Manchester.**

BCE 9480 for further information

Ceramic-lined Pumps

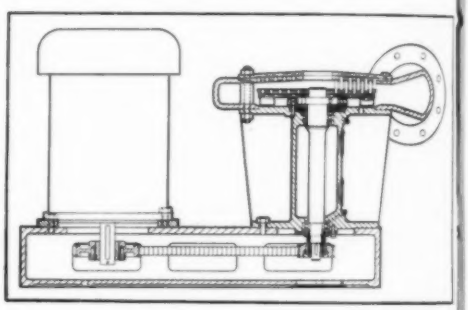
A range of ceramic-lined pumps previously manufactured by Craig Pumps Ltd. is now to be manufactured by a company know as Mitchell Craig Pumps Ltd. These remarks apply to diaphragm pumps, either hand or motor driven, which use acid-resisting rubber "Hypalon" diaphragms and centrifugal pumps which can be supplied with packed gland or mechanical seal with capacities up to 400 gpm and heads to 120 ft.

These pumps were previously mentioned in the October 1961 issue of this journal under BCE Number 8439. **L. A. Mitchell Ltd., 37 Peter Street, Manchester, 2.**

BCE 9481 for further information

Pin Disc Mill

A new series of Kek pulverisers, known as the "B" series, is now available. The grinding parts of these mills are similar to those of their gear-driven predecessors, but they have a revolutionary type of belt drive which has greatly improved their performance. Illustrated is a vertical sec-



tion of the model 3B2 mill, which covers a wide range from 600 to 3000 lb/hr. The rotating bottom disc has a diameter of 13 in. Another model, the 4B2 Kek mill, covers a range from 200 to 1000 lb/hr and has a rotating bottom disc of 7 in. The output on a given duty will depend on the physical properties of the material to be reduced and on the particle size of the powder required. Models may be fitted with an involute delivery ring, a double-outlet bin or an emulsion-type delivery ring. **Kek Ltd., Palmerston Street, Manchester, 12.**

BCE 9482 for further information

Metering Valve

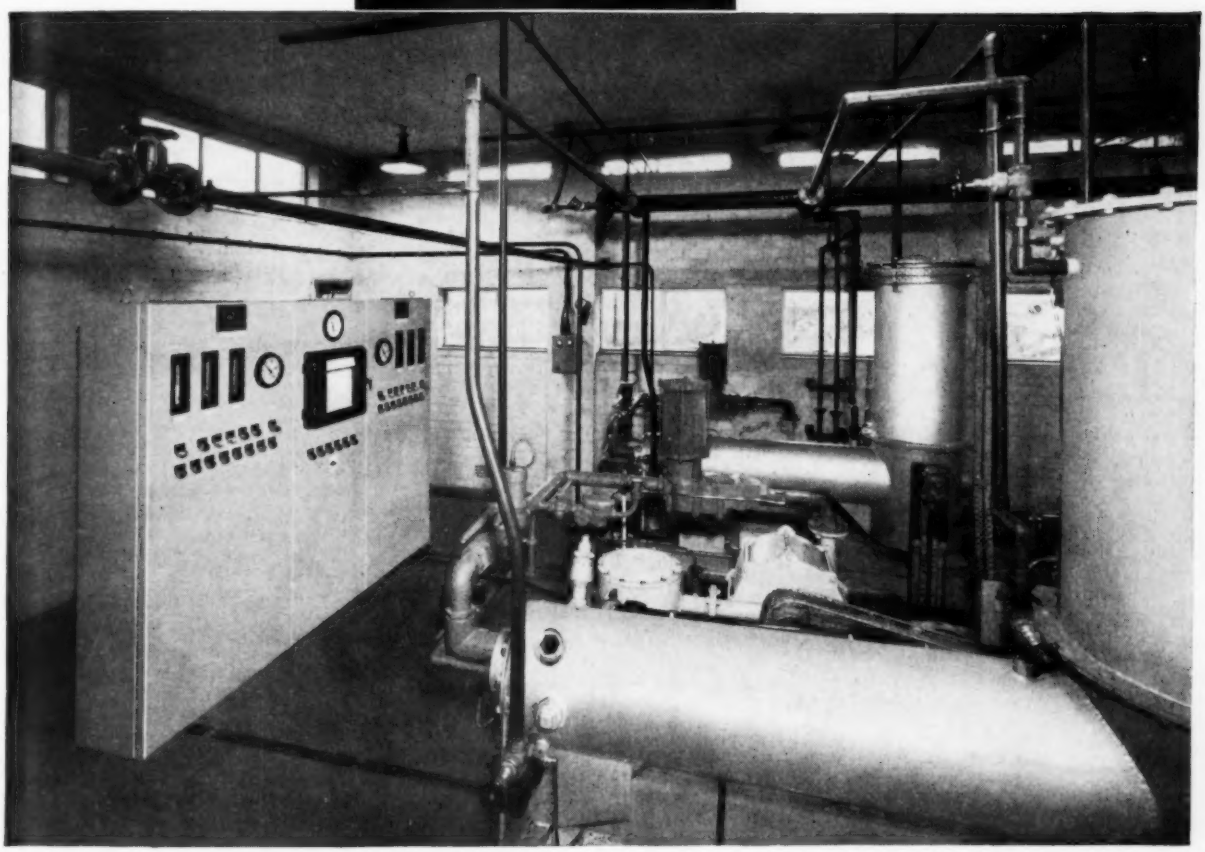
This new control makes pressure reducing or throttling devices unnecessary, and is capable of controlling volume discharge at one set rate independently of variable inlet pressures.

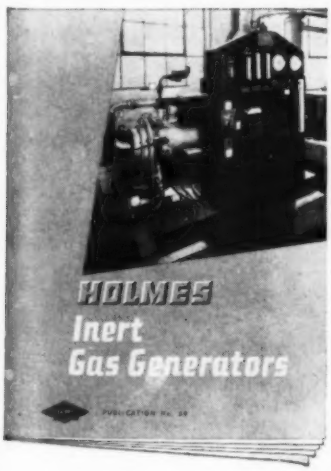
It can be used as a proportioning device and, when used with a timer and valve, will serve the same purpose as a metering pump.

Its operation depends upon orifices which are made variable by means of

BCE 9430 for further information

Holmes inert gas generators





The photograph shows the twin 2,000 c.f.h. inert gas generators installed at the Maydown Works of Messrs. Carbide Industries Ltd. The installation also includes: a central control panel, soda washers, high pressure compressors, Holmes-Kemp Dryers and a storage receiver. The inert gas, which is used for blanketing operations, is continuously monitored by a gas analyser controller to ensure that it is of a consistent high quality.

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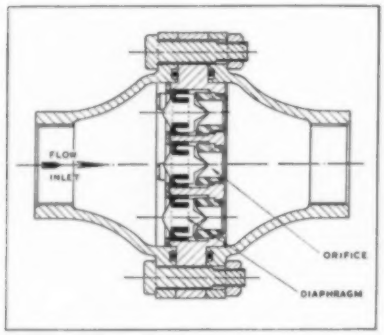
PLANT EQUIPMENT NEWS



flexible diaphragms and fixed orifices. The diaphragms, which are the sole moving parts, compensate for increased pressures by deflecting under pressure into the fixed orifices.

Size ranges at present available are from ¼ in. to 1¼ in. BSP. Maximum permissible fluid temperature and pressure are 120°C and 200 psig respectively. **Laycock Engineering Ltd., Victoria Works, Millhouses, Sheffield, 8.**

BCE 9483 for further information

Feeder for Dry Materials

Uniform feed rates can be obtained from a vibrational screw feeder which has recently become available in a range of ten sizes. The variation in material delivered is within ±1 to ±2 per cent from minute to minute, whether the material is finely divided or granular.

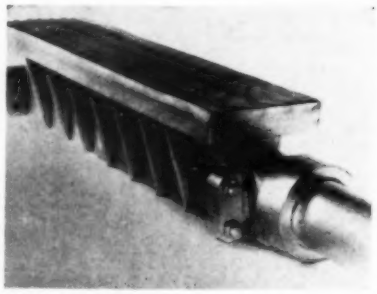
The feeder incorporates a screw mounted on a vibrator in such a way that the screw, screw trough, screw bearings and discharge tube vibrate as a single assembly while the screw is rotating.

Feed rates are proportional to the speed of screw rotation and are infinitely variable over a range of approximately 50:1. **Simon Engineering Ltd., Cheadle Heath, Stockport, Ches.**

BCE 9484 for further information

New Infra-red Burner With Controlled-wavelength Radiant Emission

This gas-fired infra-red burner is designed for high-efficiency heat transfer in low-temperature industrial processing applications. The wavelength of the



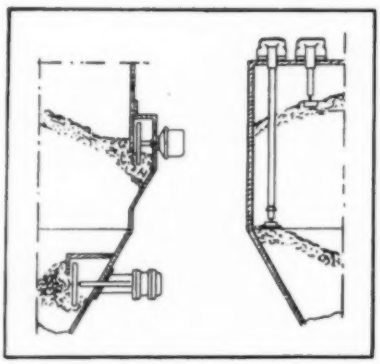
radiant heat emission provided is in the 2.0 to 4.0 micron band—the range in which most materials such as water, steel, paint, etc., have a maximum heat absorption. Thus optimum heat-transfer efficiency (maximum radiation intensity at minimum fuel input) is obtained.

Face temperature of the burner screen (4 in. × 18 in.) is 1200 to 1800°F and normal heat output is 30,000 Btu/hr. Each unit has a built-in flame adjustment, and heat pattern across an entire process assembly can be profiled or contoured to meet any demand. **Bryant Industrial Products Corp., Cleveland, Ohio, U.S.A.**

BCE 9485 for further information

Rotary-blade Level Gauges

The sensing elements of a recent design of solids level monitors consist of vanes



mounted on a shaft driven by a small electric motor. When the solids level reaches the vanes of a particular element, the rotation is hindered and the resulting torque shifts the motor and its mounting and actuates a contact which switches off the motor. When the solids level falls the vanes are able to move and a spring returns the motor to its working position, causing it to be switched on in the process.

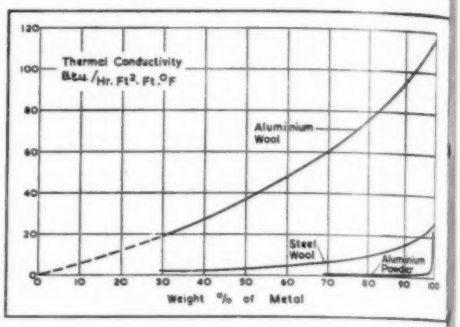
The gauges are installed in pairs to give indications of the extremes of levels in the bunkers or hoppers to which they are attached. They may be installed either horizontally or vertically. **Etablissements E. Constantin, 105 rue LaFayette, Paris, 10e, France.**

BCE 9486 for further information

Aluminium Wool

There is an increasing interest in the use of aluminium wool, when used in conjunction with polyesters and epoxy resins, as a heat-conducting medium.

Apart from its use for making moulds for thermo-plastic materials and for forming tools for deep drawing and bending of sheet metals, it is suitable for protective housings for instruments and equipment which are likely to become hot during operation.



The accompanying diagram shows the effectiveness for conducting heat of this new material which may well have applications for components used in process equipment. **Croxton & Garry Ltd., 16/18 High Street, Kingston-on-Thames, Surrey.**

BCE 9487 for further information

New Publications

One of the latest technical service bulletins (No. 1U/1) to be issued by Monsanto Ltd. is on the subject of styrene monomer. This material is now widely used in the protective coatings field in addition to being of fundamental importance in the plastics and synthetic rubber industries. The publication gives full details of the physical and chemical properties of styrene monomer, its applications, storage and handling. Sections are also devoted to health, fire and explosion hazards. A useful bibliography is given at the end of the publication.—**Monsanto Chemicals Ltd., Monsanto House, Victoria Street, London, S.W.1.**

BCE 9488 for further information

An enlarged edition of the catalogue describing the company's range of pressure and vacuum switches has been published by Londex Ltd. These switches are suitable for non-corrosive liquids and gases.

A new switch, type PS/A, is described on page 4 of the publication. This model will operate on extremely low pressures, at between ½ and 3.5 in. w.g. pressure, and, although of such high sensitivity, it incorporates heavy-duty snap-action contacts which are directly operated.

Londex comparative differential pressure switches, which operate a contact when a difference between two pressures is reached, are also described in the publication, as well as flameproof pressure and vacuum models for special applications. **Londex Ltd., Anerley Works, 207 Anerley Road, London, S.E.20.**

BCE 9489 for further information

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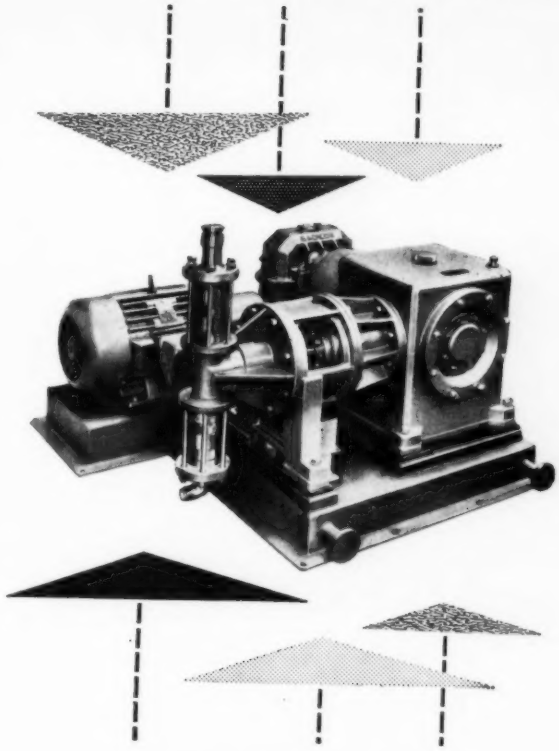

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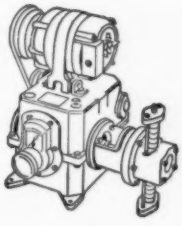

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World of Chemical Engineering

Conferences

At the U.S. National Association of Corrosion Engineers' 17th Annual Conference and 1961 Corrosion Show just concluded in Buffalo many papers of interest to the chemical industry were presented. Three entire sessions, indeed, were devoted to corrosion problems in the chemical industry.

One of the most noteworthy papers from these sessions described methods of designing corrosion out of processing equipment (R. Sanford); another dealt with gasketing problems in the chemical industry (C. M. Hobson); and an entire discussion session was devoted to tools and techniques for solving corrosion problems. Some papers dealt with specific corrosion problems and with the resistance of materials to certain media.

Within the first category came a paper on the influence of impurities on the high temperature corrosion properties of zirconium on hydrochloric acid (W. Kuhn), whereas in the latter group the corrosion of niobium and tantalum in alkaline media was discussed.

The refining industry was another to be covered (by two sessions) and included such topics as the corrosion of copper-base alloys, iron-aluminium and iron-chromium aluminium.

Protective coatings, corrosion in oil and gas processes, corrosion at elevated temperatures, cathodic protection and corrosion inhibitors were some of the other fields to receive attention at this conference.

New Plants in Brief

Polybutadiene rubber is to be manufactured at a new plant being constructed by Goodrich-Gulf Chemicals Inc. Initial annual output will be 20 million lb.

A plant for the polymerisation of polypropylene will be built at Stenungsund, Sweden, by Esso Svenska. Society Danubia Petrochemie A.G. is building a plant for the production of synthetic propylene resins near Vienna. Both these projects are licensed by Montecatini.

I.C.I. Ltd. is to spend more than £4 million on improving and developing the research, production and distribution facilities of its pharmaceuticals division. A new factory will be erected at Macclesfield, Cheshire.

Celanese Corp. of America is to build a plant costing upwards of $12 million to make acetaldehyde and 2-ethyl hexanol. The process is licensed from Aldehyd G.m.b.H., of Germany, and will produce 100 million lb/yr of chemicals by blasting air through ethylene.

New Processes

A new catalytic dealkylation process for toluene, xylenes, mixtures of the two, or alkyl benzene concentrates has been developed by Houdry Process Corp. of Philadelphia. Known as "Detol", it is a highly selective process producing close to theoretical yields of benzene. Crown Central Petroleum Corp. are to build a plant to produce 17 million gal/yr of "Detol" benzene. The process is available for licensing by Houdry.

A new process, developed by Bayer A.G., Leverkusen, for sodium borohydride ($NaBH_4$) depends upon the interaction of borax, sand and sodium at 3 atm in an atmosphere of hydrogen, and yields a stable white crystalline product. Important applications are the selective reduction of organic compounds, production of foam-like plastics, boron hydrides and many others.

Company News

Kent-Tieghi S.p.A. is the name of a new company formed in Milan by George Kent Ltd., who have acquired 51 per cent of the share capital of the Tieghi company.

Audley Engineering Co. Ltd. have changed their name to Audco Ltd. from March 1, 1961.

The Rheostatic Co. Ltd. and Black Automatic Controls Ltd. (members of Elliott-Automation Ltd.) have made joint marketing arrangements for selling certain of their electrical products.

Fertilizers & Chemicals Ltd., of Haifa, have formed two associated companies. Industrial Chemical Equipment Ltd. will design and supply general chemical equipment, and Karta Ltd. will engineer and construct cooling towers.

A carbon black factory costing £1 million is shortly to begin full production near Port Elizabeth, South Africa. Output will be about 22 million lb/yr.

I.C.I. "Terylene" will be made at a new plant at San Lorenzo, South America. Initial capacity will be 2 million lb/yr and will involve an investment of £1½ million.

A £½ million factory for the production of nylon has been opened near Cape Town by Nylon Processors of South Africa (Pty) Ltd. Full production will be 500,000 lb of yarn a year.

Personalities in the News

Mr. W. Fletcher has been appointed Sales Manager, Stainless Steel Plant, with A. Johnson & Co. (London) Ltd. He was formerly Senior Chemical and Project Engineer with Constructors John Brown Ltd., and has contributed articles to BRITISH CHEMICAL ENGINEERING.

Dr. Hugh B. Ogburn is to be Manager of the Process Engineering Department of the M. W. Kellogg Co., New York.

Major C. J. P. Ball, Chairman of Magnesium Elektron Ltd., has been awarded the Institute of Metals (Platinum) Medal in recognition of his outstanding services to the non-ferrous metal industries, and, in particular, to the magnesium industry.

Brian H. Turpin, Managing Director of Quickfit & Quartz Ltd., has been appointed Chairman of Q.V.F. Glass (Canada) Ltd.

Mr. Richard Adams has been appointed General Manager of a newly created Engineering Division of Castrol Ltd.

Mr. A. D. Lidderdale has been appointed General Manager of Leybold Elliott Ltd., the new company recently formed by Elliott Bros. of London.

Contracts

P.G. Engineering Ltd. is to build a cyclic catalytic oil gas plant consisting of two units of the Segas process, producing town gas from heavy oils.

The A.P.V. Co. is to construct for Bitmac Ltd. a complete new benzole refinery to process 8 million gal/yr of crude coke oven benzole.

Foster Wheeler Ltd. is to build a butyl rubber plant at the Esso refinery, Fawley. The new plant will cost £4½ million and produce 30,000 tons per annum.

A £60,000 contract for the supply of automatic proportioning equipment for ingredients used in the manufacture of rubber footwear has been secured by Simon Handling Engineers Ltd. from Strojimport of Czechoslovakia. This equipment will introduce closer process control and eliminate manual weighing and feeding of the mixing machinery.

R. & J. Dempster Ltd. have received an order for a plant to deal with 11 million cfd of refinery gas to be erected at Antwerp.

BCE 9433 for further information

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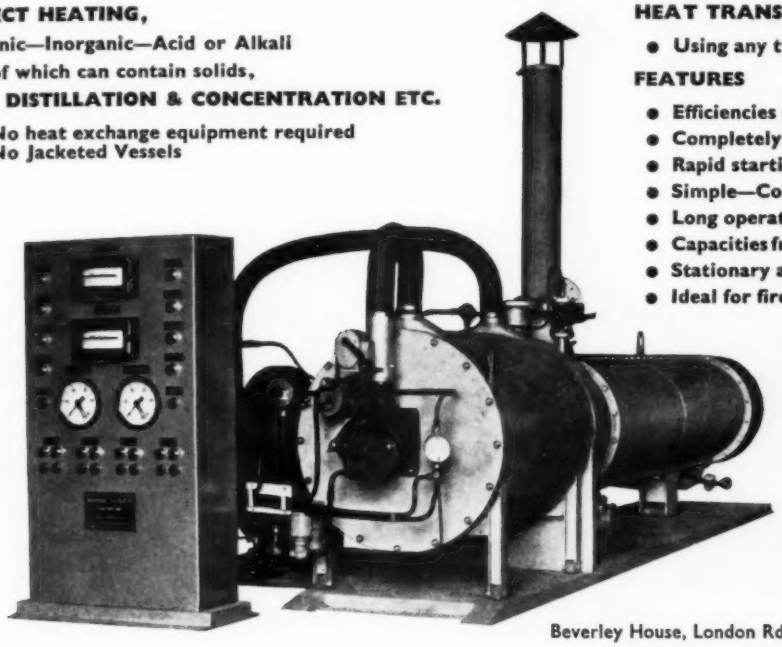

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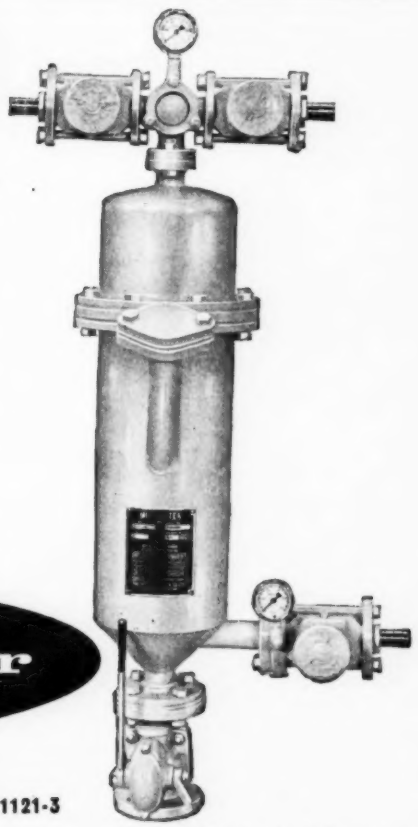

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BCE 9434 for further information

Simon-Carves Ltd. are to erect, for the West Midlands Gas Board, an Otto continuous reforming plant producing 5 million cfd of towns gas from commercial butane.

Lodge-Cotterill Ltd. have been contracted to build a scarfer fume-cleaning plant at the Newport works of Richard Thomas & Baldwin Ltd.

Whessoe Ltd. have secured a contract worth £130,000 for the supply of 17 storage tanks at Immingham Dock, Lincolnshire.

The British Oxygen Co. Ltd. have on hand the building of 19 tonnage oxygen and nitrogen plants having a value of £10½ million.

Some Problems Solved

Batch Tar Acid Distillation Column

The rotary acid distiller required a batch column and selected one with Ballart trays. The designer had to specify the product quantities and purities which would be obtained from the column, a task which required the working out of new methods for batch distillation calculations. The success of the new methods can be judged by the fact that the new performance of the reboiler follows closely that predicted. Its duty is to evaporate a 10,000-gal charge down to 500 residual gal; hence 95 per cent of the charge to the boiler still is recoverable. This has been made possible by the special design of the reboiler with its tube bundle of extremely small head-room.

The column works under vacuum and the total pressure drop from still to column head is 127 mm Hg which corresponds to 2.3 mm per actual plate.

Operation has proved quite satisfactory at rates as low as 13 per cent of the design boil-up, and the design throughput shows an overload capacity of 20 per cent without detriment to separating power.

Efficiency in practice has been found to be higher than with the corresponding bubble tray, and stabilisation time at total reflux is within 6 hours.

The column was designed and manufactured by Metal Propellers Ltd., Croydon, and is installed at Charles Lowe (Manchester) of Reddish, Stockport.

Pumping Viscous PVA Emulsions

The problem here was to rearrange a process so that, instead of small batch scale handling methods, a semi-continuous system could be used for the transfer of materials. The process involved was the manufacture of polyvinyl acetate polymer, and the main handling task was the transference of the polymer to storage. The polymerisation plant is so arranged that the polymer, which is made in 400-gal batches, has to be transferred through a distance of some 200 ft. It is at a temperature of 90°C and is fairly viscous. The new system relies upon a positive-displacement, sliding-shoe-type pump. It transfers the material through 2-in. piping to storage with a static total head of 30 ft. As soon as one complete charge of polymer has been transferred a second pump at the base of the storage tank takes over and circulates the hot material through a cooler until the polymer is at the desired storage temperature. A third pump of the same design is used for transferring blended material to road tanker. It was important that, whatever type of pump was used, it would avoid local overheating. The type chosen possesses this characteristic, since it rotates slowly. The site of the application is A. J. Smith Ltd. (Braunston, near Rugby) and the pumps were supplied by Megator Pumps & Compressors Ltd.

An Economical Electro-precipitator

Operations carried out at a limestone quarry required an electro-precipitator which had a dual function to perform: to collect dust from limestone crushers, conveyors, elevators and screens and to clean exhaust gases from the limestone driers which were fired by pitch creosote.

The precipitator was therefore constructed in two independent halves, each with its own static H.T. rectifier set, its own inlet and outlet connections, as well as fan and stack. Capital cost was reduced through the use of an economical arrangement of electrodes, without sacrifice of separating efficiency.

The modified design provides a high-efficiency separation (in excess of 99 per cent on the type of dust encountered in the above application) at a cost of 10s. per cfm and for power consumption around ½ kW per 1000 cfm.

The site of the precipitator application was the Richard Briggs limestone quarry at Clitheroe, Lancs., and the design was undertaken by Simon-Carves Ltd. (Precipitator Division).

Separation of Mycelium

One of the stages in antibiotic manufacture is the separation of the Mycelium from the culture medium. The requirements include the filtration of slurries of very low solids content and extremely clear filtrates. In addition, there must be complete freedom from contamination. Boots Pure Drug Co. Ltd., of Nottingham, have used successfully for this separation a rotary-vacuum drum-precoat filter of drum area 300 sq ft and constructed entirely of 18/8 chromium nickel stainless steel. (The makers are Davey, Paxman Ltd., Colchester.)

Digital Computers for Pipework Calculations

The design of piping systems has to take into account numerous stresses and distortions introduced by temperature and pressure, and pipe stress analysis requires a great deal of laborious calculation. The introduction of digital computers has made this work much easier and such a machine can be programmed to perform all the work previously carried out manually. The calculation of moments, deflections along the piping, forces on constraints and so on is then completely automatic.

As an example, the calculations for a system which had ten anchor points, two re-entrant loops and 23 branches was solved in four hours of computer time. This problem and others of a similar kind was handled by the Postal Computing Service of the English Electric Computing Bureau at Kidsgrove, Staffordshire.

Infra-red Analyser for Operator Safety

Although not strictly a chemical plant application, the use of an instrument of this kind in the motor industry is worthy of note.

The aim was to ensure that the atmosphere inside a special low-temperature engine test room should not have a harmful concentration of CO. An instrument was installed for giving an alarm when the atmosphere attained 400 ppm which is tolerable for periods of up to 30 min, the duration of engine tests. The instrument has a range up to 1000 ppm and can give a visual and audible alarm at any concentration within that range. Accuracy of indication is ±1 per cent of full scale.

The instrument was supplied to the Ford Motor Co. by Mine Safety Appliances of Glasgow.

Control of Bulk Pumps

Evershed & Vignoles Ltd. have recently installed an extremely safe system for controlling the loading of hazardous petrochemicals into road tankers. The installation to which this system was applied requires products to be transferred in varying quantities, and for this reason pumps and pipelines have been split into product groups in order to provide flexibility of the pumping capacity.

The road tanker remote-loading control equipment is intrinsically safe, and one of its important features is the anti-static earthing of the tanker to be loaded. This ensures that a product cannot be fed into the tanker until it is confirmed that the correct earthing has been established.

The maintenance of pressure is the main criterion for the pump control system. When loading of a tanker commences, the pressure in the system drops and this brings into operation a pressure switch which controls the pump motor starter. The pressure is then restored to the required level. The increased flow rate arising from operation of the pump is monitored by means of a flow transmitter and the output from the latter actuates an alarm relay which, in conjunction with a follow-up relay unit, keeps the pump operating independently of the pressure switch.

On completion of a loading operation the driver closes a valve in the tanker loading arm, and the pressure switch, in combination with a timing device, keeps the pump running for about 2 min in order to maintain the correction pressure in the line. Comprehensive automatic shut-down facilities are provided.